NASA-CR-54269

# POTASSIUM CORROSION TEST LOOP DEVELOPMENT

## Quarterly Progress Report No. 5
## For Quarter Ending October 15, 1964

EDITED BY E. E. HOFFMAN



prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2547




SPACE POWER AND PROPULSION SECTION
MISSILE AND SPACE DIVISION
GENERAL ELECTRIC
CINCINNATI, OHIO 45215

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# POTASSIUM CORROSION TEST LOOP DEVELOPMENT

## QUARTERLY PROGRESS REPORT 5

Covering the Period

July 15, 1964 through October 15, 1964

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SPACE POWER AND PROPULSION SECTION
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# POTASSIUM CORROSION TEST LOOP DEVELOPMENT

## I INTRODUCTION

This report covers the period from July 15, 1964 to October 15, 1964 of a program to develop a prototype corrosion test loop for the evaluation of refractory alloys in boiling and condensing potassium environments which simulate projected space electric power systems. The envisioned prototype test consists of a two-loop Cb-1Zr facility; sodium will be heated by direct resistance in a primary loop and will be used in a heat exchanger to boil potassium in the secondary corrosion test loop. Heat rejection for condensation in the secondary loop will be accomplished by radiation in a high-vacuum environment. The immediate corrosion test design conditions are shown below; it is expected that the temperatures could be increased by about 400°F when testing is extended to include refractory alloys stronger than Cb-1Zr.

1. Boiling temperature, 1900°F
2. Superheat temperature, 2000°F
3. Condensing temperature, 1350°F
4. Subcooling temperature, 800°F
5. Mass flow rate, 20 to 40 lb/hr
6. Vapor velocity, 100 to 150 ft/sec
7. Average heat flux in the potassium boiler - 50,000 to 100,000 BTU/hr $ft^2$

The development program is proceeding with the construction and operation of three Cb-1Zr test loops, each of which will be used in a sequence of component evaluation and endurance testing. Loop I, a natural convection loop, has been operated for 1,000 hours with liquid sodium at a maximum temperature of 2260° to 2380°F to evaluate the electrical power vacuum feedthroughs, thermocouples, the method of attaching the electrodes, the electrical resistivity characteristics of the heater segment, and the use of thermal and electrical insulation. Loop II, a single-phase sodium, forced-circulation loop to evaluate the primary loop EM pump, a flowmeter, flow control and isolation valves, and pressure transducers has operated for 1,985 hours at the end of the current reporting period. This loop which operates with a pump inlet temperature of 1985°F will be terminated after 2,500 hours operation. The Prototype Corrosion Test Loop, a two-loop system, design of which has been completed, will include a boiler, turbine simulator, and condenser in addition to the above components. This facility will be used to develop and endurance test (2,500 hours) the components required to achieve stable operation at the corrosion test design conditions.

The quarterly reports issued for this program will summarize the status of the work with respect to design considerations, construction procedures, and test results. The topical report on Loop I is expected to be released in February, 1965. Detailed topical reports will also be issued to describe the Loop II and the Prototype Loop tests. Additional topical reports will be prepared to cover such areas as materials specifications, purification of potassium and sodium, and inert gas purification and analysis.

## II PROGRAM STATUS

### 1. Loop II Operation

Loop II has operated 1,985 hours since its startup at 1000 July 2, 1964, at the following test conditions:

Heater outlet temperature - 2050°F

Pump inlet temperature - 1985°F

Metering valve temperature - 650°-800°F

Loop pressure - 144 psia

Sodium flow rate - 415 lb/hr

Electrical power input - 6.75 KW

Vacuum chamber pressure - $8.3 \times 10^{-9}$ torr

During this period two scheduled shutdowns and four unscheduled shutdowns by activation of the loop safety circuits have occurred and are summarized in Table I. Test operation during the quarter is presented chronologically below.

The first unscheduled shutdown of the loop occurred at 1130 July 19, due to the over-temperaturing of a thermal overload relay in the EM pump power supply. The over-temperature of the thermal relay occurred on an excessively hot day when the ambient temperature of the test cell, where the EM pump power supply is located, exceeded 100°F. The thermal relay has been adjusted to its maximum temperature setting to permit higher ambient operating temperatures, and a large capacity exhaust fan is now in use to increase the laboratory ventilation and reduce the possibility of a shutdown due to activation of the thermal overload relay.

A scheduled shutdown of the loop was made at 0800 August 3, to determine and correct the cause of progressive deterioration in the metering valve actuation system and to identify and locate the source of a dark deposit which had accumulated on the colder regions of the test chamber wall.

The dark deposit had concentrated in 1-1/2-inch wide bands which traced the path of the water cooling channels. Darkening appeared to increase slowly to a maximum intensity during the first 200 to 300 hours of the test and then remain relatively constant. During the bakeout following the unscheduled shutdown of July 19, the deposits completely disappeared from the heated chamber walls but reappeared during the subsequent steady state operation in the exact location of the water cooling channels of the vacuum chamber. The tendency of the deposit to migrate in the chamber indicated that the material had a relatively high vapor pressure at the chamber bakeout temperature of 400°-500°F. During the scheduled shutdown of August 3, the loop temperature was decreased to 1000°F

TABLE I

LOOP II - OPERATION HISTORY

July 2 - October 15, 1964

| Time | Date | Test Hours | |
|---|---|---|---|
| 1000 | July 2 | 0 | Loop startup |
| 1130 | July 19 | 409 | Unscheduled shutdown - over-temperature of EM pump thermal overload relay |
| 1330 | July 20 | | Resume test |
| 0800 | August 3 | 734 | Scheduled shutdown - metering valve modified by substituting L-605 bushing and stem for original parts - chamber wall deposits analyzed - thermocouples repaired |
| | August 18 | 734 | Attempt to resume test - metering valve and flow difficulties |
| | August 18 | 734 | Scheduled shutdown - Saginaw ball nut and stem substituted for L-605 stem and stem bushing in metering valve |
| 1300 | August 22 | | Resume test |
| 0610 | August 23 | 757 | Unscheduled shutdown - defective ion-pump cell |
| 1300 | August 24 | | Resume test |
| 1930 | August 24 | 763 | Unscheduled shutdown - argon instability in ion pump |
| 1430 | August 25 | | Resume test |
| 0900 | September 10 | 1154 | Unscheduled shutdown - argon instability in ion pump |
| 2000 | September 15 | | Resume test |
| 2400 | October 15 | 1985 | Test continuing to 2,500 hours |

and the chamber walls were heated to approximately 500°F to allow the deposit to concentrate on a cooled coil which was located near the center of the vacuum tank. This coil was installed initially so that it might be used to collect sodium in the event that a leak developed in the loop. The inlet air to the coil was cooled to approximately -20°F by passing it through a Ranque-Hilsch Vortex Tube*. The substance deposited on the collector coil was identified as cadmium. Several possible sources, including bolts, nuts, and electrical connectors, have been checked for cadmium coatings but no source of cadmium has been established.

Several defective thermocouples were also repaired during the August 3-17 test shutdown. Shorts and breaks in the W-3%Re legs of the thermocouples were the principal problem.

During this shutdown, examination of the valve actuation system revealed that the actuation problem originated from galling in the threaded section of the valve stem and bushing. Self-welding of the highly stressed threads was initially considered to be a potential problem and both stem and bushing had been gold plated. Upon disassembly of the valve, it was observed that the valve stem and bushing had bonded and, after the initial break-away, could be operated only with a significantly higher operating torque than had been required prior to the start of the test. The increased operating torque in turn resulted in extremely rough operation of the gear drive assembly which exhibited a tendency to bind at higher torque levels and so further increased the torque required to actuate the valve.

In an attempt to operate the valve, which normally required 1-2 inch-lbs of torque to either open or close, approximately 6 ft-lbs were exerted. Repeated application of this abnormally high torque level resulted in rotating the valve body. This movement resulted in momentary contact of the bracket with the Cb-1Zr foil insulation on the lower loop heater coil. Arcing from the electrically charged heater to the grounded stainless steel gear bracket resulted. Fortunately, the damage as shown in Figure 1 was confined primarily to the metallic foil used to insulate the heater thermally, and only two small pits less than 0.010-inch in diameter and less than 0.002-inch deep were found on the surface of the heater tube. The pits were easily removed by light polishing of the tube wall with alumina paper. Chemical analyses performed on acid smear samples taken from the surface of the polished area indicated that no stainless steel constituents were present on the Cb-1Zr tube. Restraining bars were subsequently welded from the support structure to the gear bracket assembly to prevent a recurrence of this type of movement.

Two approaches were initiated to eliminate the valve actuation problem. The first approach was simply to replace the gold-plated stainless steel stem and aluminum bronze bushing with similar L-605 (Co-base alloy) parts. An exploded view of the valve was shown in a previous report of this program[1]. The second approach considered was the replacement of the entire threaded stem and stem bushing with a Saginaw** ball bearing screw assembly which would require less operating torque and possibly minimize the operational problem due to galling and self-

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* Fulton Cryogenics, Cincinnati, Ohio 45224

1 Potassium Corrosion Test Loop Development, Quarterly Progress Report No. 4, For Period Ending July 15, 1964, NASA Contract NAS 3-2547, NASA-CR-54167.

** Saginaw Steering Gear Division, General Motors Corp., Saginaw, Michigan.

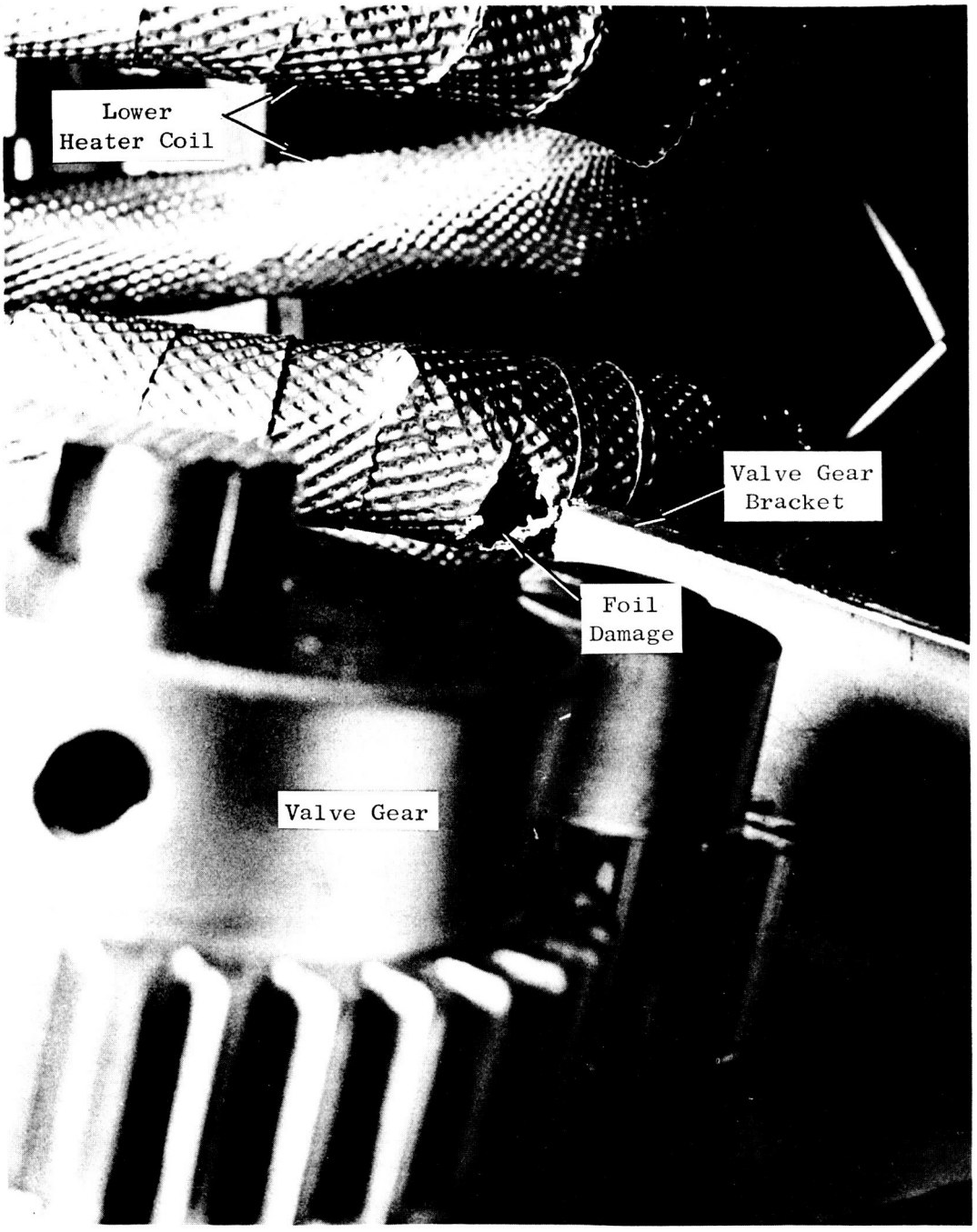



Figure 1. Local Damage to Metallic Foil Thermal Insulation on Loop II Heater Coil Caused by Electrical Arcing to Valve Gear Bracket Rotated During Test Operation. The Damage was Observed After 734 Hours. (C64080513)

welding. Although the ball bearing screw approach was believed to be the better solution of the problem, it was decided that the L-605 stem and bushing would be evaluated first since these materials were on hand and no modification to the valve drive assembly would be required. Following machining, the L-605 threaded surfaces were work hardened to minimize the galling tendencies of these valve parts. The procurement of the Saginaw ball bearing screw and the valve modifications were initiated to be available as immediate replacements for the threaded stem and bushing in the event that the substitution of L-605 parts did not eliminate the problem. A distinct advantage of the Saginaw ball bearing valve stem drive approach is the ease of substitution of commerically available cermet or ceramic balls for the standard hardened stainless steel balls.

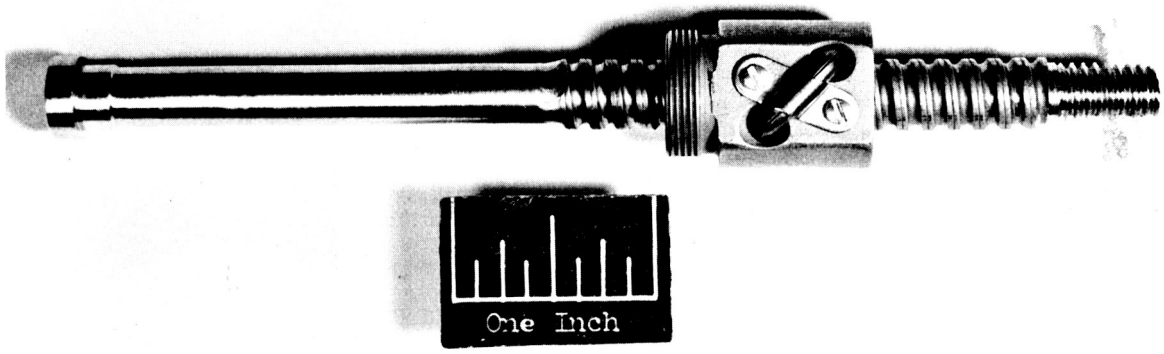
All modifications and repairs were completed on August 18 and the chamber was evacuated and baked out. Some flow instabilities were observed when the test was being brought to temperature, but steady forward flow was established. However, binding in the metering valve actuation system was encountered and the test was shut down to replace the L-605 stem and stem bushing with the Saginaw ball nut and stem shown in Figure 2.

On August 21 the chamber was evacuated and baked out, and on the following day the test was resumed. The metering valve performed satisfactorily with the Saginaw ball bearing screw assembly.

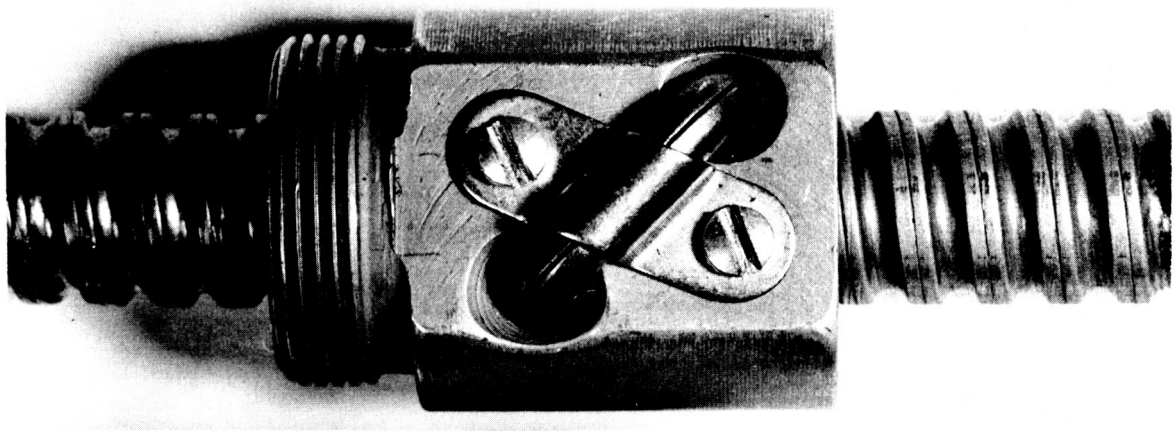
During test startup, attempts to circulate the sodium showed a low indicated sodium flow rate for a given EM pump power setting as established from previous runs. A check on the accuracy of the flowmeter reading by a thermal balance across the heater showed an abnormally high temperature rise for a given power input which indicated that the low flow reading was correct.

Several attempts to increase the flow by jogging the EM pump power to dislodge the apparent obstruction were not successful. A high sodium flow rate, however, was established and maintained by reversing the electrical power leads to the EM pump and operating the loop in the reverse direction. The electrical power leads to the EM pump were returned to the normal hook up and flow was established in the correct flow direction with the flow rate for a given EM power setting comparable to previous test conditions. The loop was then dumped in an attempt to collect the apparent obstructing material in the surge tank. Steady flow in the forward direction was established following refilling of the loop. Additional information on the apparent plugging problem should be obtained by careful examination of the loop components and the sodium following completion of the 2,500-hour test.

On August 23, after 757 hours of accumulated test time, the test was shutdown by activation of the argon flooding safety circuit which is triggered by an increase in the getter-ion pump current. A check of the vacuum chamber showed that one of the four pump cells had developed a short which increased the ion pump current and closed an overpressure relay which fired the explosive valve, flooding the chamber with argon gas. The rapid rise in chamber pressure resulting from the argon flooding turns off the ion gauge filament and an accessory 110-volt outlet which activates a relay that shuts off the power to the loop heater and the EM pump.




(a)



(b)

Figure 2. Saginaw Ball Nut and Valve Stem Used in the Modification of the Loop II Metering Valve. (a) Assembly, (b) Enlarged View of Ball Nut.

The temperatures of various regions of the loop during the cool down period following actuation of the protective argon flooding system are given in Table II. All of the loop components, with the exception of the fairly massive EM pump (23 pounds of Cb-1Zr), cooled to a temperature of less than 1000°F in less than 4 minutes.

The test was resumed on August 24 by pumping with the three sound pump cells after electrically isolating the defective getter-ion pump cell. An argon instability occurred in the getter-ion pump at 1930 on August 24. The resultant surge in getter-ion pump current (equivalent to a pump pressure of $5 \times 10^{-5}$ torr) was sufficient to activate the associated safety circuits and shut down the test. While restarting the test on August 25, surges in the getter-ion pump current were noted and simultaneous partial pressure scans for argon confirmed that rapid releases of argon from the pump were responsible for the pressure excursions in the system. Instabilities of this type are not uncommon in diode-type getter-ion pumped systems[2]. Calibration of the partial pressure analyzer[3] with argon and flooding of the test chamber with argon as cited above, no doubt, contributed to the instabilities noted on several occassions during loop operation.

Loop test conditions were obtained on August 25 and the test continued. During the 400 hours of uninterrupted loop operation, the metering valve continued to show the apparent plugging tendency described earlier. This valve has been operated repeatedly during the test to increase the flow in the bypass line of the loop in an attempt to maintain the valve temperature at 800°F. Despite the repeated adjustments to increase the flow rate, the valve temperature slowly decayed from the initial temperature of 800°-900°F to 640°F in several days, which is essentially the equilibrium temperature of loop components not in contact with the 1900°-2000°F liquid metal in the main loop. The 640°F temperature is indicative of very low or no flow in the bypass line. The exact cause of the temperature decay has not been established although the effect has been repeatedly observed. A typical temperature decay of the valve temperature after an adjustment is shown in Figure 3. The valve temperature was permitted to exceed the 800°F design test temperature to more clearly demonstrate the decay. Within approximately 48 hours following adjustment of the metering valve, the valve temperature had dropped to 700°F, indicating essentially no flow in the bypass line.

A typical valve adjustment showing the relative movement between the plug and the valve seat as the stem is rotated is shown in Figure 4. The valve in its initial position with the plug firmly seated and with no flow in the bypass line is at an equilibrium temperature of 640°F. The valve stem is then rotated 17° in the second position which moves the valve plug 0.006-inch and withdraws the

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2 Barrington, Alfred E., High Vacuum Engineering, Prentice-Hall, Inc., Englewood Cliffs, N.J., 1963, p. 101.

3 Potassium Corrosion Test Loop Development, Quarterly Progress Report No. 4 for Quarter Ending July 15, 1964, p. 43.

TABLE II

TEMPERATURES OF VARIOUS LOOP II COMPONENTS FOLLOWING ACTUATION OF PROTECTIVE ARGON FLOODING SYSTEM

| Time Minutes | Temperature, °F | | | |
|---|---|---|---|---|
| | EM Pump Outlet | Bottom Heater Coil | Heater Outlet | EM Pump Inlet |
| 0 | 1930 | 1840 | 2000 | 1957 |
| 1 | 1692 | 1390 | 1408 | 1426 |
| 2 | 1627 | 1123 | 1242 | 1114 |
| 3 | 1563 | 1011 | 1077 | 993 |
| 4 | 1508 | 947 | 965 | 900 |
| 5 | 1472 | 909 | 871 | 823 |
| 10 | 1344 | 919 | 634 | 603 |
| 20 | 1077 | 725 | 561 | 485 |
| 30 | 890 | 603 | -- | -- |

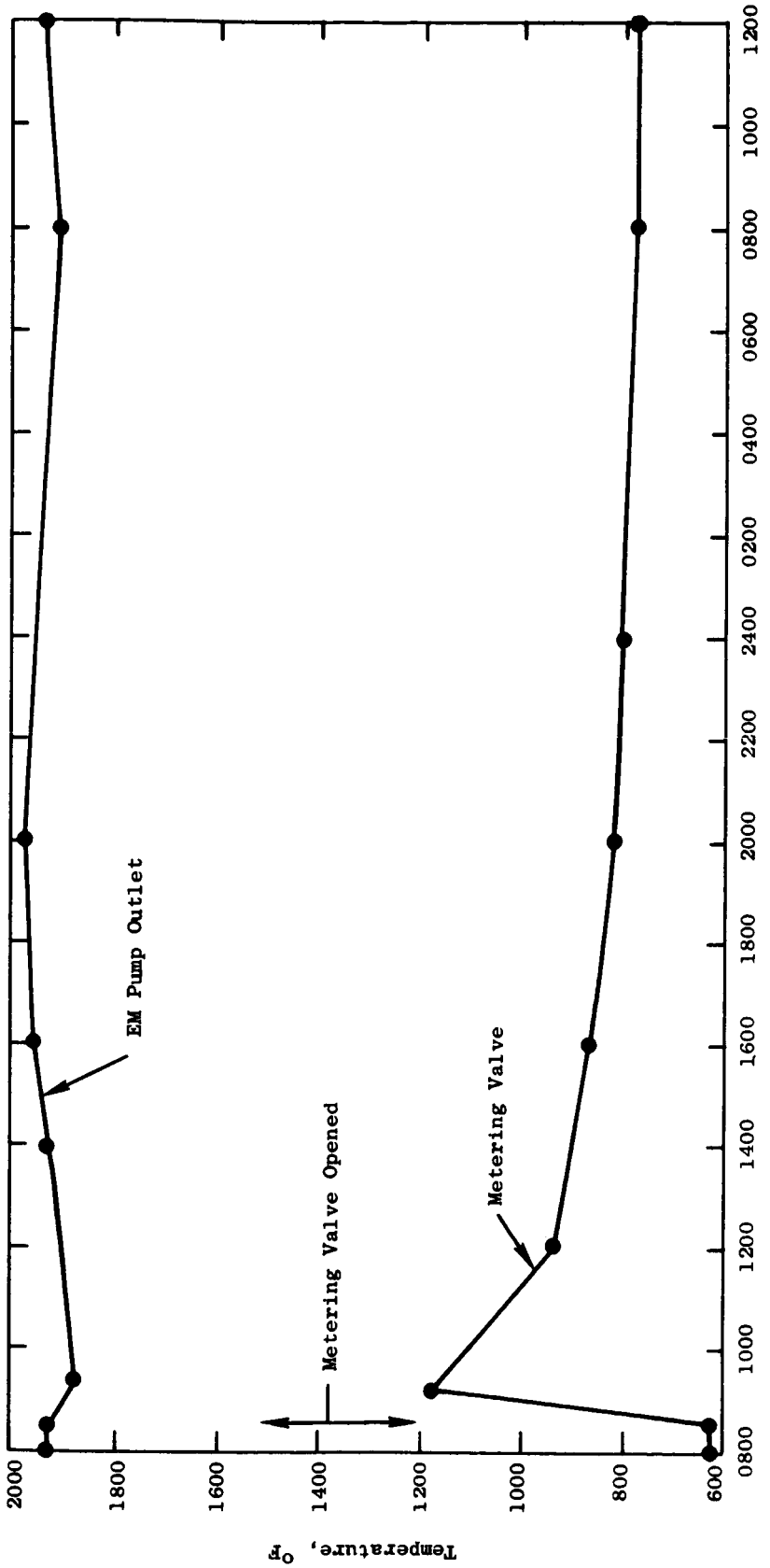
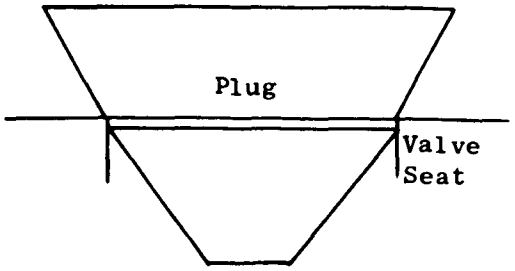
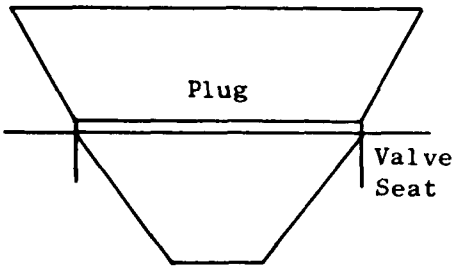



Figure 3. Temperature Decay of Loop II Metering Valve After a Valve Adjustment During Steady State Operation.



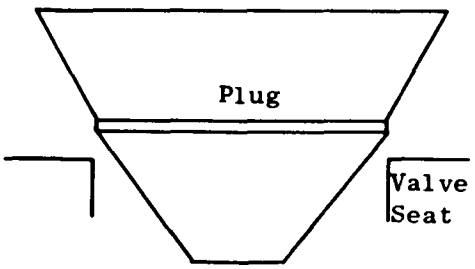

Initial Off Position

Plug Position - Fully Closed Valve
Temperature - 640°F




Second Position

Stem Rotation - 17°
Plug Position - 0.006 in. Axial Lift
Flow Gap - Zero
Flow Area - Zero
Valve Temperature: 640°F




Third Position

Stem Rotation = Additional 28°, Total of 45°
Plug Position = 0.014 in. Axial Lift
Flow Gap = 0.0043-Inch
Flow Area = 0.002-Inch$^2$

| Valve Temperature °F | Time, Hours |
|---|---|
| 1175 | 0 |
| 930 | 4 |
| 800 | 16 |
| 780 | 24 |
| 770 | 32 |
| 744 | 56 |
| 693 | 80 |
| 671 | 104 |
| 644 | 148 |

Figure 4. Summary of a Typical Adjustment of Loop II Metering Valve in Attempt to Maintain Valve Body Temperature at 800°F During 2000°F Steady State Operation.

cylindrical section of the plug between the metering cone surface and the seating surface of the plug from the valve orifice. In this position the flow area is essentially zero and the valve temperature remains unchanged. In the third position, the valve stem is rotated an additional 28° which raises the plug an additional 0.008-inch for a total lift off of 0.014-inch from the initial off position. The gap between the plug and seat is 0.0043-inch which is equivalent to a flow area of 0.002 square inch and allows sufficient sodium flow through the bypass line to raise the temperature of the valve body from 630° to 1175°F. The drop in valve temperature after various time periods following opening of the metering valve is also given in Figure 4.

The test was restarted on September 10 and continued, uninterrupted, throughout the remainder of the quarter, accumulating 1,985 hours of test operation on October 15. A typical argon instability observed during this period is shown in Figure 5. The sudden pressure rises associated with the argon release, which are shown, were not of sufficient magnitude to activate the safety circuits.

A plot of the Loop II test chamber pressure from the beginning of loop operation on July 2 until the end of the quarter is given in Figure 6. The details of the six test shutdowns have been discussed above. The total pressure and partial pressures of various gaseous species for the Loop II test chamber environments during 1,800 hours of test operation are given in Figure 7. It may be noted that the $N_2$ or CO(m/e=28), Ar(m/e=40), $H_2O$(m/e=18), and possibly $H_2$ (m/e=2) are the principal species present after 1,800 hours of operation. Unfortunately, a malfunction in the ion source of the partial pressure analyzer at high accelerating voltages has prevented scanning for hydrogen for the last 1,000 hours of loop operation. Comparison of the sum of the partial pressures with the total pressure indicates that the hydrogen pressure could be in the range 2-4 x $10^{-9}$ torr after 1,800 hours of loop operation.

2. Loop II Sodium Pressure Measurements

A Taylor Instrument Company electronic volumetric pressure gauge (slack diaphragm) and a refractory alloy, stressed diaphragm, fast response transducer supplied by Consolidated Controls Corporation and modified by General Electric are being evaluated in Loop II.

The volumetric pressure transducer consists of a diaphragm housing, diaphragm, NaK filled capillary, and bourdon tube with all parts which are wet by the process fluid (sodium in Loop II, potassium in the Prototype Loop) made from Cb-1Zr alloy. The gauge has a pressure range of 0-150 psia and a maximum temperature rating of 2000°F with a response time of one second for 63% of the range. The gauge has operated successfully for 1,985 hours as of October 15, and a precise calibration will be made at the completion of the 2,500-hour test.

The stressed diaphragm pressure transducer which has a response of up to 100 cps was designed and fabricated for evaluation in Loop II to supplement the (1 cps) slack diaphragm pressure transducer in measuring pressure fluctuation during periods of unstable boiling and condensing which may be encountered in the Prototype Loop. The stressed diaphragm transducer consists of a pressure housing with a

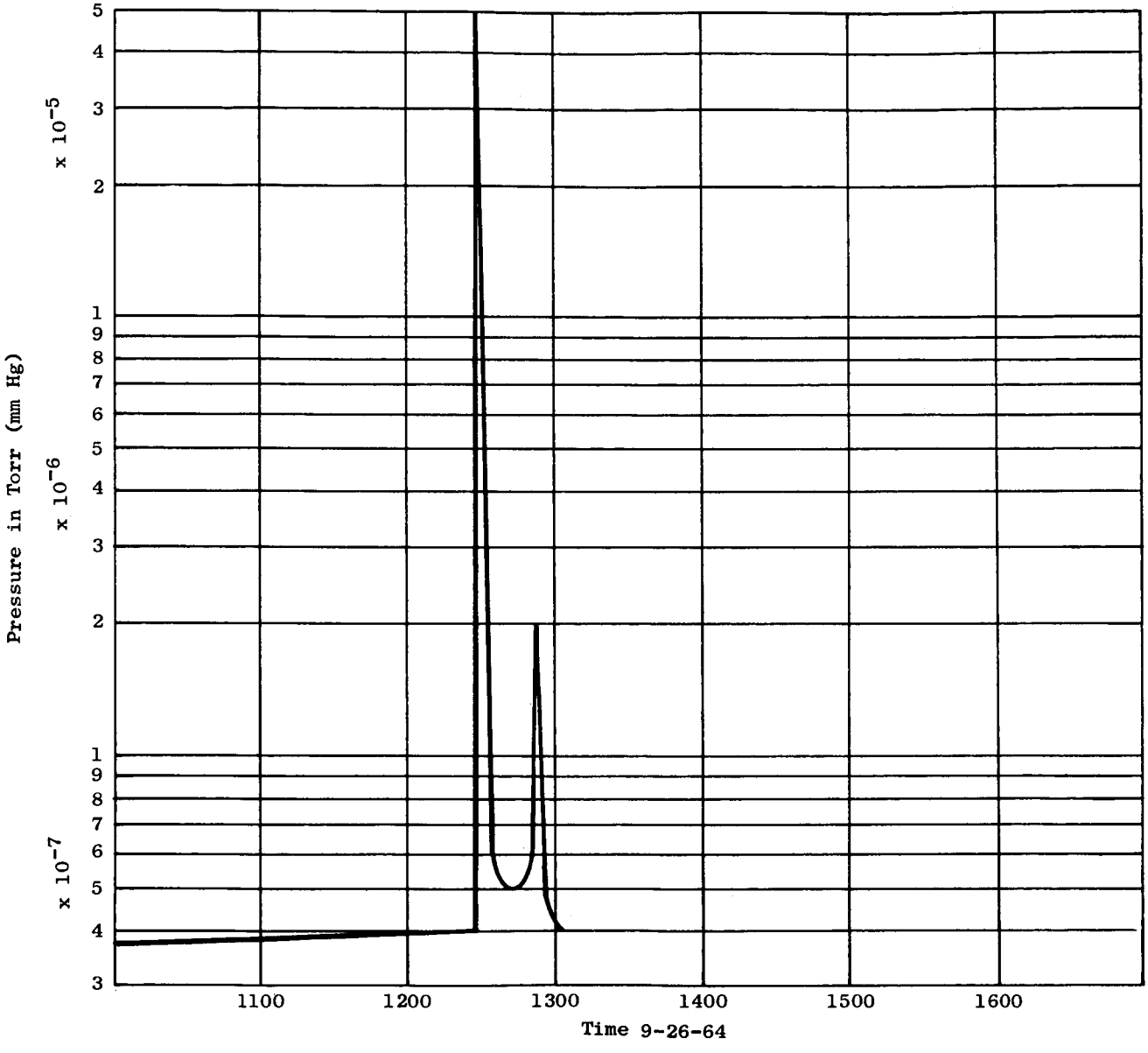



Figure 5. Loop II Vacuum Chamber Total Pressure During an Argon Instability in the Ion Pump.

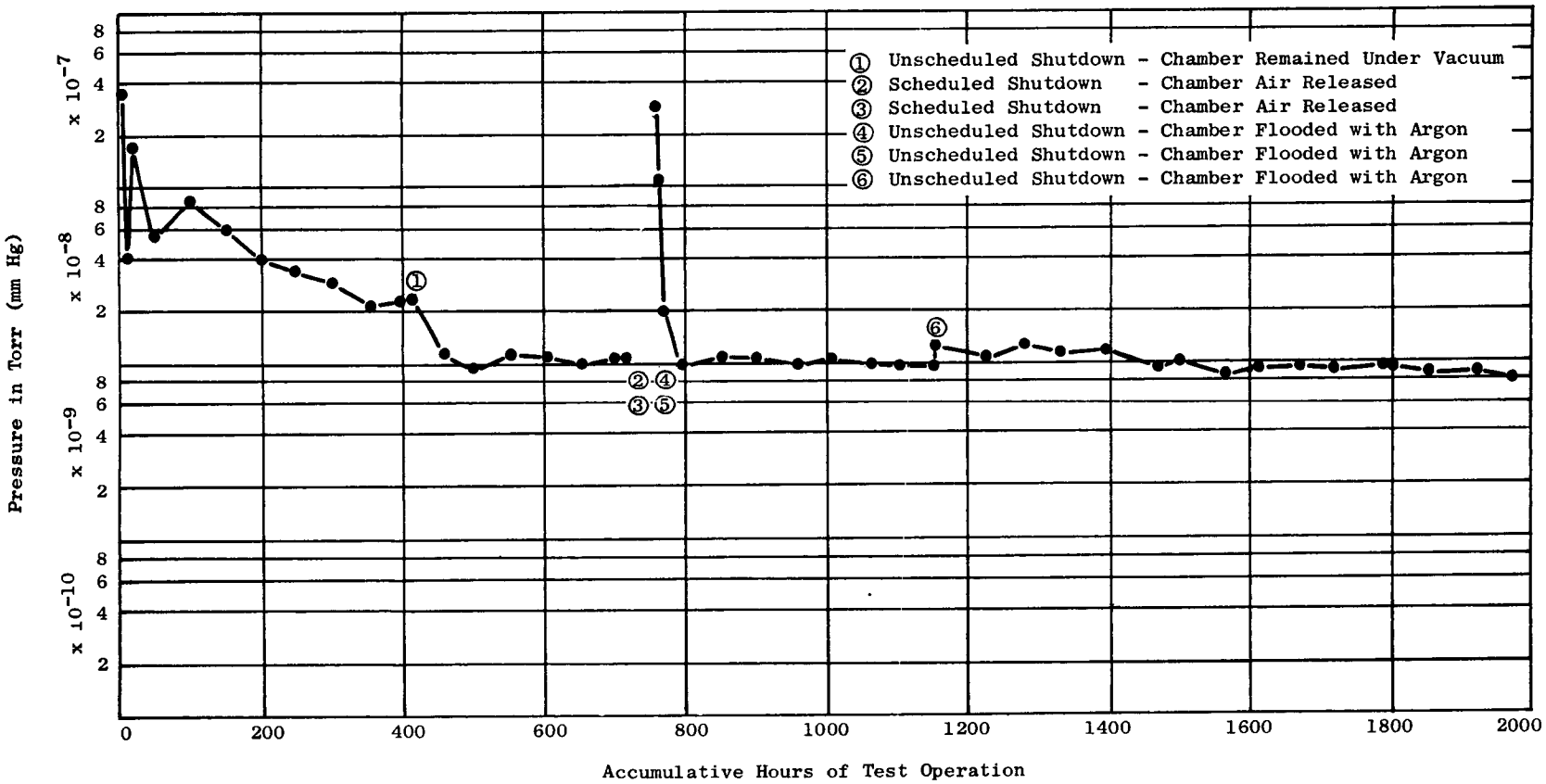



Figure 6. Loop II Test Chamber Pressure During Loop Operation
July 2 - October 15, 1964.

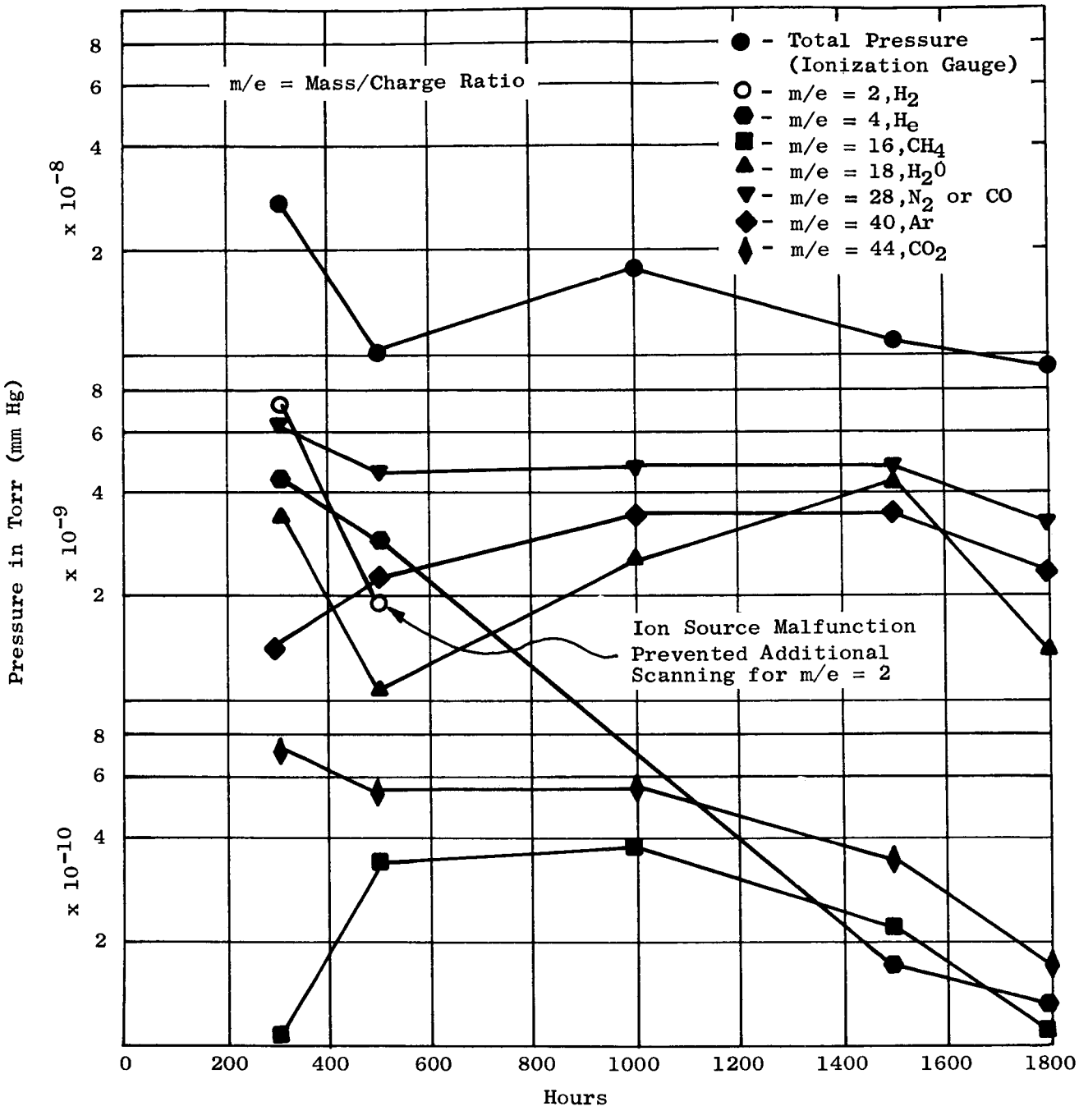



Figure 7. Total Pressure and Partial Pressures of the Various Gaseous Species in the Loop II Test Chamber vs. Total Hours of Loop Operation.

0.015-inch thick T-111 alloy diaphragm which serves as a stressed member. Deflection of this diaphragm, which is a function of the pressure on it, is the measured quantity.

An initial calibration was performed on the diaphragm assembly before the 2200°F heat treatment of the assembly welds. During the annealing of the Cb-1Zr process tube, the stressed diaphragm pressure transducer was located outside the annealing furnace and did not exceed 1500°F during this annealing treatment. The results of this calibration indicated that the diaphragm operated satisfactorily over the full pressure range of 0-150 psia and was repeatable within 0.5% over four full scale pressure cycles. The net change in the output level for a 0-150 psia change in pressure was approximately 100 millivolts.

A second calibration was made with the transducer installed in Loop II but prior to filling the loop with sodium. The calibration which was made with argon gas at room temperature showed that the net change in output for the 0-150 psia change in pressure was 52 millivolts which was repeatable to within 0.53% over four complete pressure cycles or approximately one-half of the output measured in the initial calibration of the same pressure range. The decrease in output can probably be attributed to a change in the diaphragm deflection characteristics during the heat treating of the Cb-1Zr assembly welds after the initial calibration. During the 1-hour 2200°F annealing treatment, the temperature of the diaphragm housing did not exceed 1500°F.

A third calibration was performed with the loop filled with sodium during the bakeout period with the transducer temperature of 480°F. During this calibration, there was a net zero shift of approximately 12% of the span. During loop operation, a continued zero shift in the positive direction was observed, indicating creep in the diaphragm, although the slope of the output characteristics curve remained unchanged. During the shutdown of August 3 after 784 hours of operation, the output changed from 290.5 mv to 219.9 mv in reducing the loop pressure from 150 psia to 0 psia, or a net change of 70.6 mv compared to 52 mv for the calibration before test operation.

On August 26, 1964, after 800 hours of test operation, the transducer failed to respond to pressure changes in the loop. It is possible that the loss of signal may have been caused by breakage of electrical leads inside the chamber due to a.c. induced vibration. An examination of the transducer will be made upon completion of the 2,500-hour loop test in November to determine the cause of the loss of response and the positive zero shift of the transducer.

3. Alkali Metal Sample Vacuum Distillation Unit

A high vacuum distillation unit to separate alkali metal from an non-volatile species which might be present in a filter sample is shown in Figure 8. This unit will be used to remove up to 50 grams of sodium from a 5-micron in-line filter cup which will be used to filter the sodium from Loop II when it is discharged from the system following completion of the 2,500-hour test. The purpose of the filtering procedure is to trap and concentrate any mass transfer crystals which may be

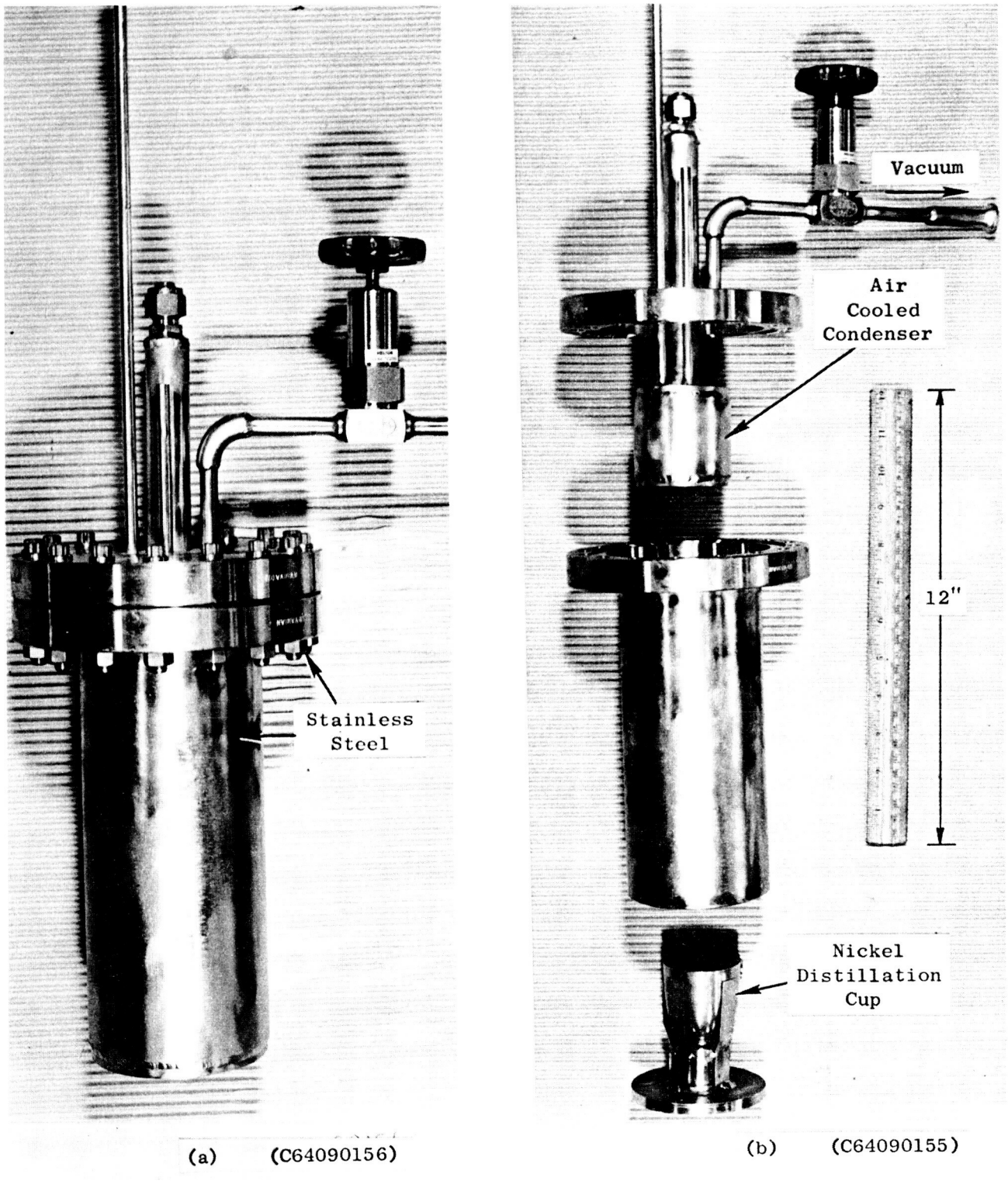



Figure 8. High Vacuum Alkali Metal Distillation Unit. (a) Assembled Unit. (b) Exploded View of Components.

present in the sodium. Sodium and potassium from the Prototype Loop will also be filtered in this manner, and the filter cups will be decontaminated of alkali metal in the unit shown. The opening of the filter cup and loading of the distillation unit will be performed in a vacuum-purge, controlled atmosphere chamber.

4. Prototype Loop Fabrication

Fabrication of the primary and secondary EM pump ducts continued with the completion of all required machining and welding of the process tubes. The interference fits between the finned ducts and the pipe duct will be obtained by the following procedure: (a) heating the pipe duct to 500°F in air, (b) chilling the finned duct in liquid nitrogen, and (c) removing the finned duct from the liquid nitrogen and rapidly inserting it into the pipe duct. This procedure proved satisfactory during the manufacture of the Loop II EM pump duct after an initial attempt involving only heating of the outer wrapper to 1200°F in an argon environment resulted in seizing. It has been suggested that freezing out of moisture from the air on the finned duct may act as lubricant or barrier to galling during insertion of the chilled finned duct into the heated duct pipe. An additional consideration is the lower temperatures of both parts relative to the earlier procedure which should also reduce the tendency for galling to occur.

The six Taylor pressure transducers were received during this report period. The sample of NaK taken by Taylor during the filling of these transducers has been received. Analyses of this sample produced 3.3 ppm oxygen in a 3.268-gram sample and 4.3 ppm oxygen in a 3.353-gram sample for a 3.8 ppm oxygen average. Additional analyses on the NaK used to fill the transducers gave the following results: less than 1 ppm-Mn, Mg, Cu, Be, Ag, Sr, Ca; less than 2 ppm-Ba; less than 5 ppm-Fe, B, Co, Al, Sn, Pb, Cr, Ti, Ni, Mo, V; less than 10 ppm-Si, Zr.

The machining procedures required for manufacture of the turbine simulator nozzle assembly were established. A 32-rms surface finish in the nozzle throats was produced by the Elo-polish process, a refinement of electric spark discharge machining. A final polish of the nozzle throat with one-micron alumina will be used to produce an 8-rms surface. The Mo-TZM alloy blades for this sub-assembly were received from the vendor and orders were placed for the remainder of the machined components.

Orders were also placed for all Cb-1Zr alloy components which require machining or forming operations with the exception of the condenser sub-assembly. It is anticipated that all components will be placed on order during the next report period.

Oven panels for the sodium transfer system and the dolly for the sodium purification system have been received. Installation of these items is in progress. The sodium purification system should be installed and ready for use on the Prototype Loop in about one month. All parts for the 50-pound potassium hot trap have been received and fabrication procedures have been initiated. Most parts and components for the potassium still, transfer systems, argon-vacuum manifold, and samplers have been received.

5. Alkali Metal Purification and Handling System for the Prototype Loop

Preliminary drawings (48) of the potassium purification and alkali metal handling and sampling systems were completed and reviewed. Revisions are in progress. The potassium purification and loop filling system for the Prototype Loop is shown in Figure 9. A summary of the various steps in the purification and transfer procedure is given in Table III.

6. Tungsten/Rhenium Thermocouple Wire Studies

W-3%Re/W-25%Re was selected as the basic thermocouple alloy combination for instrumenting all alkali metal loops in the Potassium Corrosion Test Loop Development Program. The selection was based on its compatibility with the test loop alloy (Cb-1Zr), its high and fairly linear thermal emf, and its reported excellent stability over extended periods of time in vacuum at elevated temperatures. Recent studies by Hendricks and McElroy[4] yielded results which indicate that tungsten-rhenium base thermocouples attached to the surface of columbium yield a more stable emf than platinum-rhodium base thermocouples attached in this manner.

Although various tungsten-rhenium alloy thermocouple combinations have been used for a number of years, a national standard has as yet not been adopted. Matched tungsten-rhenium thermocouple wire of various alloy combinations, however, can be purchased with a specified accuracy to tentative calibration tables adopted by the thermocouple wire suppliers. The policy adopted for this program is to purchase wire to a chemical or material specification and conduct the calibration test at General Electric on the thermocouple wire in accordance with the over-all quality control plan. The W-3%Re wire is purchased from the Lamp Metals Components Department of the General Electric Company and the W-25%Re wire is purchased from Hoskins Manufacturing Company. All thermocouple wire used in a specific loop test is supplied from one matched lot with its own calibration.

The W-3%Re alloy was selected as the positive leg of the thermocouple in preference to pure tungsten because of its improved handling properties relative to unalloyed tungsten, resulting in considerably less breakage during installation and operation. Even with the increased ductility of the W-3% wire over unalloyed tungsten wire, fractures of the W-3%Re leg of thermocouples, particularly near the brazed joint between the thermocouple wire and the nickel vacuum feedthrough tube,[5] have been one of the chief sources of thermocouple difficulties during the installation and operation of Loops I and II of this program. Studies to improve this situation are discussed later in this section.

The results of the calibration of the thermocouple wire used in instrumenting Loop II are shown in Figure 10. The calibration test was originally run in an argon atmosphere at 5 psig using a General Electric-Advanced Technology Laboratory certified Pt/Pt-10%Rh thermocouple as the calibration standard. The test was not run in a vacuum environment, initially, because a suitable high vacuum calibration facility was not available at that time. The test was later repeated in the 2000°F to 2200°F range in a Brew vacuum furnace capable of $1 \times 10^{-6}$ torr. The vacuum

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[4] Hendricks, J. W. and McElroy, D.L., High-Temperature High-Vacuum Thermocouple Drift Tests, ORNL-TM-883, August 1964.

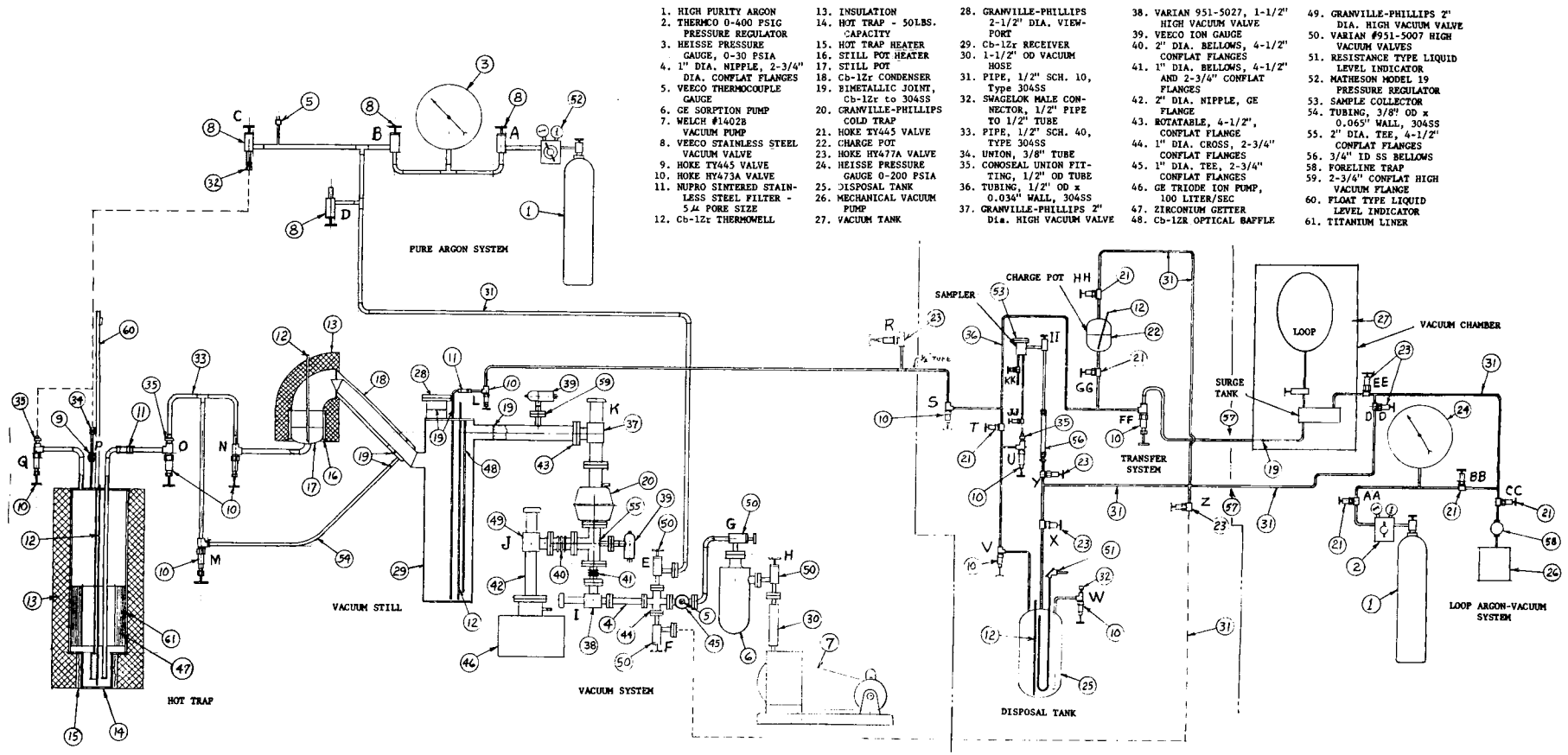


Figure 9. Potassium Purification and Loop Filling System For the Prototype Loop.

## TABLE III

### POTASSIUM PURIFICATION AND FILLING PROCEDURE FOR THE PROTOTYPE LOOP

1. Outgas and helium leak check all parts of the apparatus. The maximum allowable outgassing rate is 5 micron-liters per hour at 500°F. The maximum allowable leak rate is 5 x $10^{-10}$ std. cc of air per second.
2. Transfer 50 pounds of potassium from the shipping container to the hot trap(14) through a 5-micron stainless steel filter at a temperature of 200°-240°F.
3. Outgas the potassium under vacuum at a temperature of 450°F until the pressure decreases to a constant value.
4. Hot trap the potassium at 1300°-1400°F for 50 to 100 hours.
5. Reduce the temperature to about 240°F and obtain a sample of the potassium through valve O (sampler not shown in schematic). Analyze the sample for oxygen, carbon, and metallic impurities.
6. Clean valve O and connect transfer tubing at O.
7. Bring valves O, N, M, the transfer lines, still pot(17), condenser(18), and receiver(29) to about 240°F. Fill the still pot through valve M, thus filling the annulus between the inner and outer condenser tubes with potassium.
8. Vacuum distill the potassium at a temperature between 500° and 550°F while maintaining the lower portion of condenser and the receiver below 300°F. Refill the still pot intermittently through valve N.
9. Close valves F, G, and J and pressurize the still with about 5 psig argon through valve E.
10. Heat the transfer lines and the transfer system to about 240°F and close valves FF and HH.
11. Transfer potassium into the charge pot(22) to valve HH and Z.
12. Close valve S and open valve V and transfer the charge into the disposal tank(25) by pressurizing through valve HH. This cleans the transfer system with potassium.
13. Close valve V, refill the charge pot and take a sample through valves T, U, and JJ. Analyze the specimen for oxygen.
14. Pressurize the potassium from the charge pot into the surge tank through valves GG and FF. The transfer is complete when a rise is noted on the pressure gauge(24).
15. Close valve FF and evacuate the potassium loop through valves EE and CC.
16. Pressurize the surge tank through valves AA, BB, and EE.
17. Circulate the potassium in the loop at 500°F.
18. Evacuate the transfer lines through the disposal tank and return the potassium to the surge tank.
19. Take a sample of potassium and transfer the remainder to the disposal tank. Analyze the sample for oxygen.
20. Refill the charge pot and take another sample. Analyze for oxygen and metallic impurities.
21. Transfer the potassium to the loop surge tank prior to starting loop test.

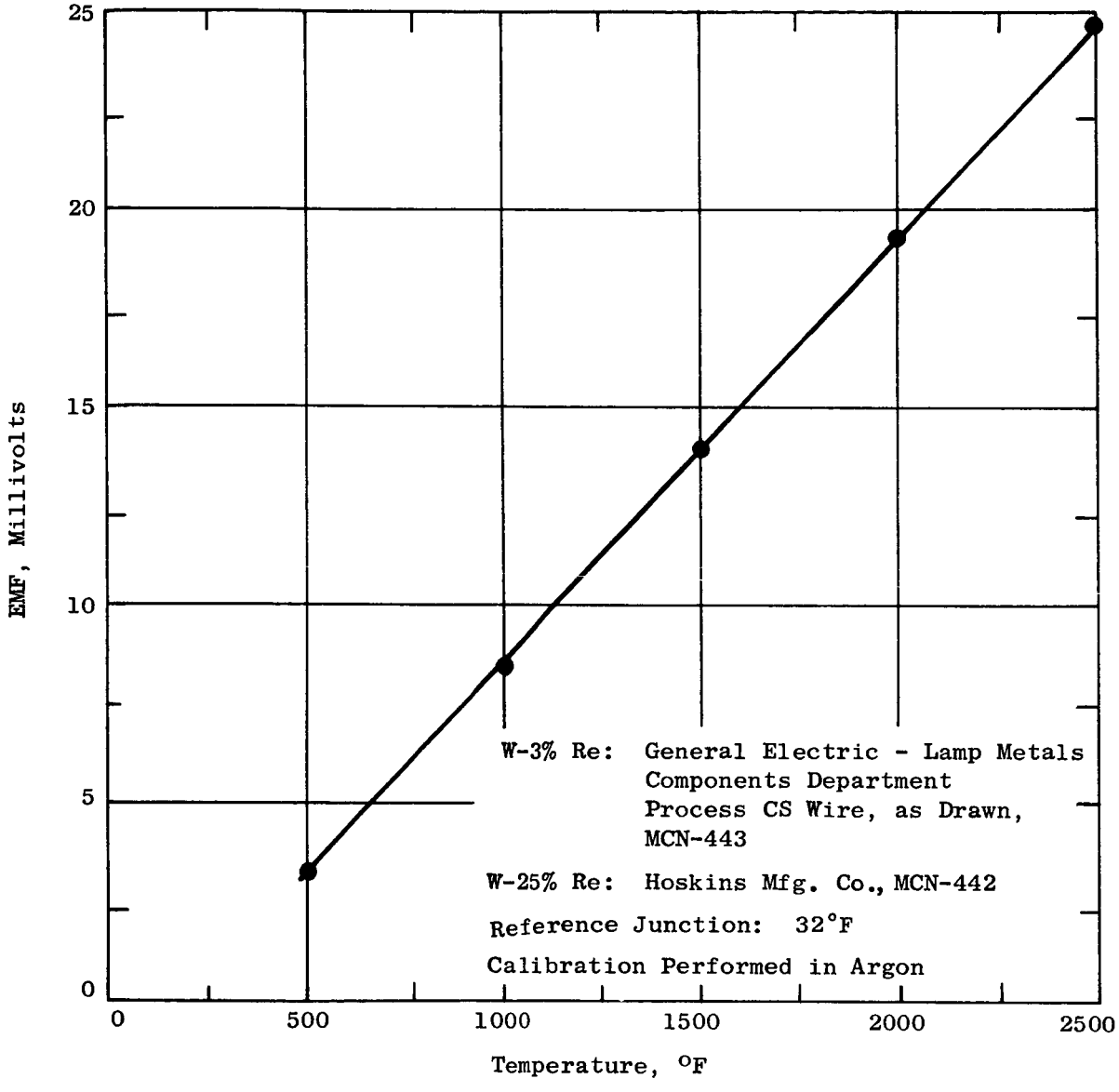


Figure 10. Thermal EMF of W-3% Re/W-25% Re Thermocouple Wire for Loop II.

calibration was in good agreement with the original argon calibration as can be seen in Figure 11. Similar calibration tests of tungsten-rhenium thermocouples in both inert gas and vacuum environments have been reported[6] as in general agreement to about 4000°F. However, all future calibration tests will be conducted in a vacuum environment with a total pressure less than 1 x $10^{-7}$ torr.

As mentioned earlier, breakage of the 5-mil W-3%Re leg of thermocouples has been a problem in the instrumentation and operation of tests to date. In order to improve the handling characteristics of this material, an evaluation study was initiated in cooperation with the Lamp Metals Components Department of General Electric to determine the optimum annealing temperature for the W-3%Re w wire. The reverse bending characteristics as well as the emf output of one lot of wire as a function of heat treatment were included in the study. The wire was evaluated following annealing at 2280°F, 2460°F, 2820°F, 3180°F and in the "as-drawn" condition.

The results of the bend tests are given in Table IV. Since failures of the W-3%Re leg were most common near the brazed joint which seals the thermocouple wire in the nickel tubulation of the thermocouple feedthrough, several combinations of brazing alloy and heat amperage used to make the joint were studied also. The results indicated that the wire annealed at the highest temperature (3180°F) would tolerate the greatest number of 90 degree reverse bends prior to fracture. The results obtained on the brazed samples indicated that the lower melting Premabraze 615 alloy and the lower heater amperage used to make the braze seal had the least effect on the fracture tendencies of the W-3%Re wire.

The thermal emf characteristics of the various W-3%Re legs as a function of heat treatment were studied by calibrating combinations of the W-3%Re wire with a common W-25%Re junction vs. a calibrated Pt/Pt-10%Rh thermocouple. The test was conducted in a $10^{-5}$ torr vacuum environment. The results of this test are shown in Table V and indicate that the emf of the W-3%Re/W-25%Re thermocouple increases with higher annealing temperature for the W-3%Re leg over the range investigated. At 1984°F, the emf of the thermocouple with the "as-drawn" W-3%Re leg was 0.45 mv less than the thermocouple with the leg annealed at 3180°F. This difference is equivalent to approximately 45°F at this temperature level. These results suggest that the higher annealing temperatures tend to stabilize the wire and, therefore, the wire annealed at the higher temperature should be less likely to drift during the 2,500-hour loop test.

The 3180°F annealing temperature was designated for the entire lot of W-3%Re wire to be used in instrumenting the Prototype Loop based on the results of the bending and emf tests cited above.

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[5] Potassium Corrosion Test Loop Development, Quarterly Progress Report #4, covering the period April 15, 1964 to July 15, 1964, NASA Contract NAS3-2547, NASA-CR-54081, p. 27.

[6] Hoskins Mfg. Co., Tungsten-Rhenium Thermocouple Alloys.

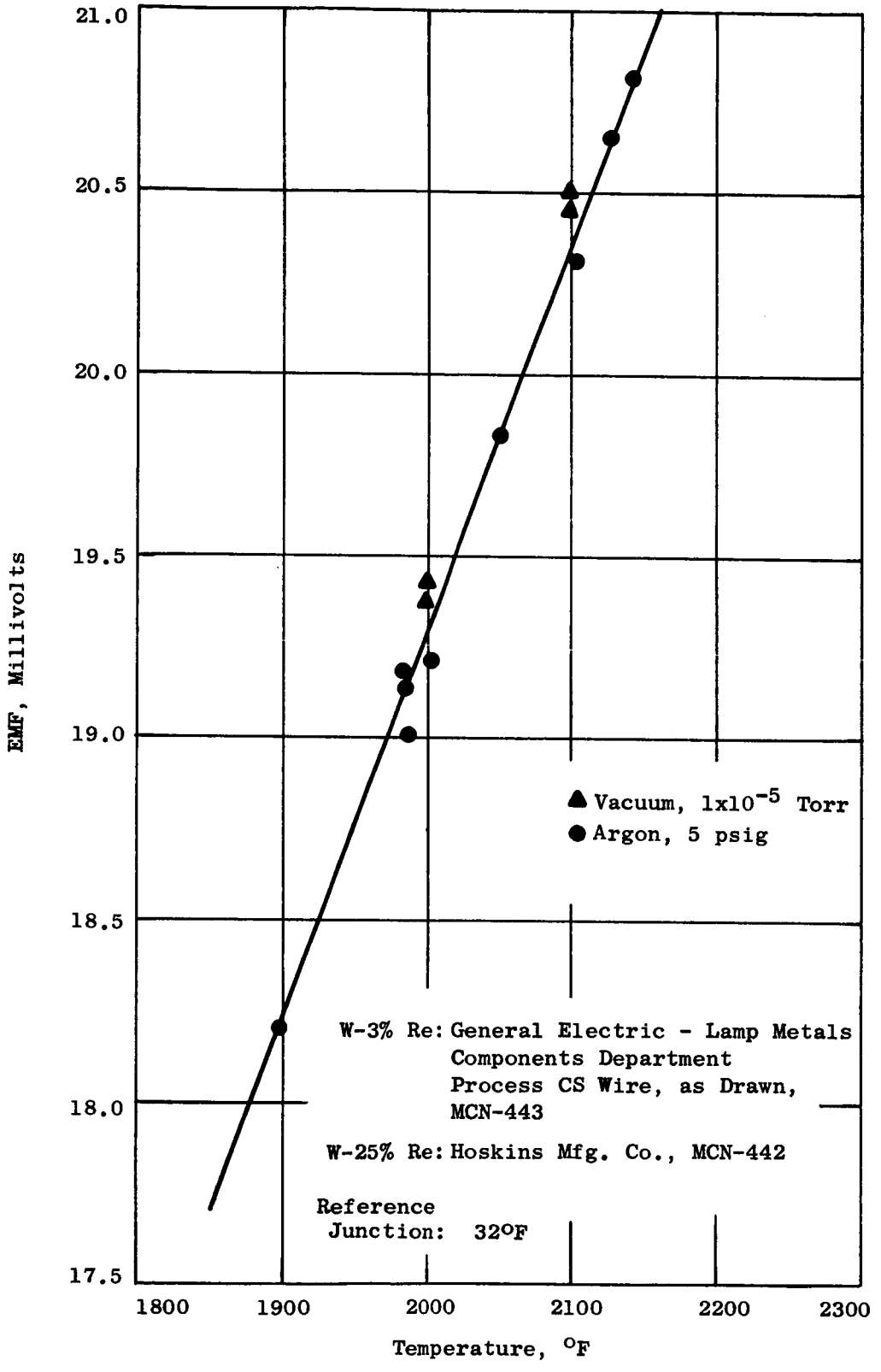


Figure 11. Comparison of Vacuum Calibration of Loop II W-3% Re/W-25% Re Thermocouple Wire with Argon Calibration.

TABLE IV

RESULTS OF BEND TESTS ON TUNGSTEN-3% RHENIUM WIRE FOLLOWING VARIOUS HEAT TREATMENTS AND BRAZING OPERATIONS

| | Number of 90°-2T Reverse Bends[a] Required to Fracture 0.005-Inch Wire | | | | |
|---|---|---|---|---|---|
| | CS[b] | CLS[c] (2280°F) | CLS (2460°F) | CLS (2820°F) | CLS (3180°F) |
| 1. Wire | 6 | 10 | 8 | 11 | 11 |
| 2. Wire Brazed[d] with Premabraze 615[e], 12 Amps | 9 | | 7 | | 11 |
| 3. Same as 2., but 18 Amps | 7 | | 6 | | 7 |
| 4. Wire Brazed with BT Alloy[f], 21 Amps | 5 | | 6 | | 9 |
| 5. Same as 4., but 28 Amps | 3 | | 6 | | 6 |

a. Average of four tests (minimum) on each specimen. Bending of the wire of the brazed specimens was confined to the wire immediately adjacent to the brazed joint.

b. General Electric Lamp Metals Components Department designation, CS - Chemically cleaned and straightened.

c. General Electric Lamp Metals Components Department designation, CLS - Chemically cleaned annealed, and straightened. The normal General Electric - LMCD annealing temperature is proprietary.

d. Brazed into 0.050-inch OD x 0.005-inch wall nickel tubes in an argon environment using a contact resistance heater.

e. 61.5Ag - 24.0Cu - 14.5In, Melting Point - 1155°F.

f. 72Cu - 28Ag, Melting Point - 1435°F.

TABLE V

EFFECT OF ANNEALING TEMPERATURE OF W-3%Re WIRE ON THE EMF OUTPUT OF A W-3%Re/W-25%Re THERMOCOUPLE IN $10^{-5}$ TORR VACUUM. (ALL W-3%Re WIRES FUSED TO A COMMON W-25%Re WIRE WITH 32°F REFERENCE JUNCTION)

| Block Temperature[b] | CS[c] | Annealing Temperature of CLS[a] W-3%Re Wire | | | |
|---|---|---|---|---|---|
| | | 2280°F | 2460°F | 2820°F | 3180°F |
| 75°F | 0.224 mv | 0.230 mv | 0.232 mv | 0.236 mv | 0.239 mv |
| 519°F | 3.984 | 4.072 | 4.106 | 4.152 | 4.183 |
| 940°F | 8.150 | 8.276 | 8.325 | 8.399 | 8.445 |
| 1476°F | 13.885 | 14.068 | 14.152 | 14.252 | 14.323 |
| 1984°F | 19.500 | 19.613 | 19.730 | 19.848 | 19.949 |
| 2205°F | 21.780 | 21.982 | 22.102 | 22.232 | 22.342 |

a. General Electric Lamp Metals Components Department designation, CS - Chemically cleaned, annealed, and straightened. The normal General Electric - LMCD annealing temperature is proprietary.

b. Measured by standard Pt/Pt-10%Rh thermocouple.

c. General Electric Lamp Metals Components Department designation, CLS - Chemically cleaned and straightened.

Five thermocouples fabricated from this wire and the W-25%Re wire purchased from Hoskins Mfg. Company have been calibrated. Three of the five thermocouples were made by spot welding the wires directly to a Cb-1Zr sheet similar to the split-junction thermocouple as used in Loop I and II instrumentation. The other two thermocouples were of the beaded junction type where the thermocouple wires are spot welded together and normally used as a well thermocouple. The thermocouple assembly mounted in the vacuum flange ready for installation in the vacuum furnace is shown in Figure 12-A with the ice bath reference junction and the thermocouple selector switch. An enlarged view of the thermocouple bundle is shown in Figure 12-b. The calibration test station is shown in Figure 13. Not shown is the cylindrical molybdenum thermal block which is positioned in the furnace and into which the thermocouple assembly is inserted to minimize any temperature gradients in the vicinity of the hot junction. The results of the calibration test of the Prototype Loop thermocouple wire are shown in Figure 14. The maximum deivation between all five thermocouples was less than 10°F and no significant difference could be detected between the thermocouples made by spot welding the individual wires to the Cb-1Zr sheet and the beaded thermocouples which were not in contact with the Cb-1Zr sheet.

## 7. Refluxing Potassium Compatibility Tests

The two refluxing potassium capsule tests which will be conducted to determine the extent of mass transfer of Mo-TZM alloy tubular insert specimens located in the condenser region of Cb-1Zr alloy capsules were described in an earlier report[7].

During the past quarter the test facility has been completed and the capsules loaded with potassium. The tests will be started early in the next quarter.

One of the refluxing capsules is shown in Figure 15 prior to being installed in the test facility. Two Cb-1Zr alloy sheet specimens, 0.5-inch wide x 0.080-inch thick x 3.5-inch long, were placed in the liquid zone and five tight-fitting, 1.0-inch long tubular insert specimens of Mo-TZM alloy were placed in the condensing region. Weight, dimensional and chemical changes on both the Cb-1Zr and the Mo-TZM specimens will be determined following test. The Mo-TZM inserts are held in position in the condensing region by Cb-1Zr support pins plug welded into the capsule wall. In order to increase the emittance of the condensing zone, a very fine thread was machined into the OD surface of this region of the capsule as shown in Figure 15. This technique was used in preference to a previously used procedure of grit blasting the surface with $Al_2O_3$. It was found that residual $Al_2O_3$ particles embedded in the surface of the Cb-1Zr resulted in high oxygen concentrations in these regions[8] which made it impossible to determine the extent of oxygen contamination which might occur during the refluxing test.

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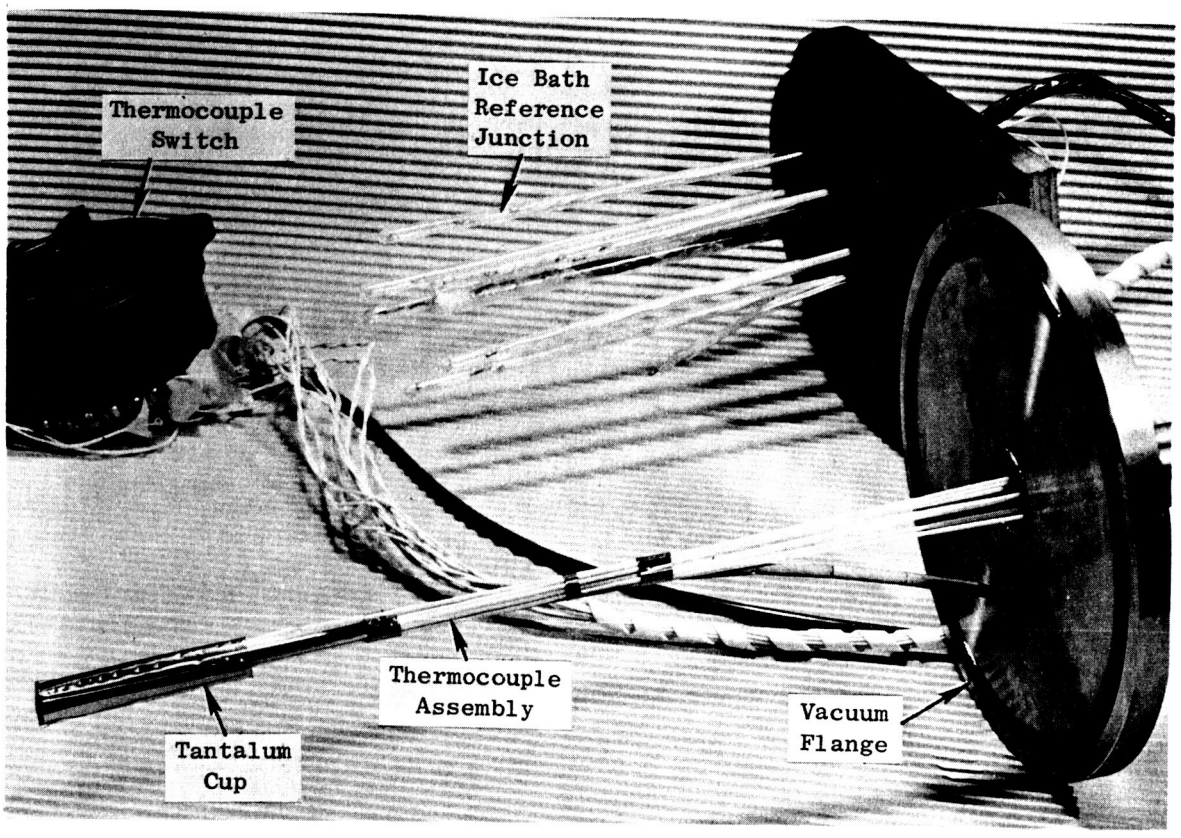
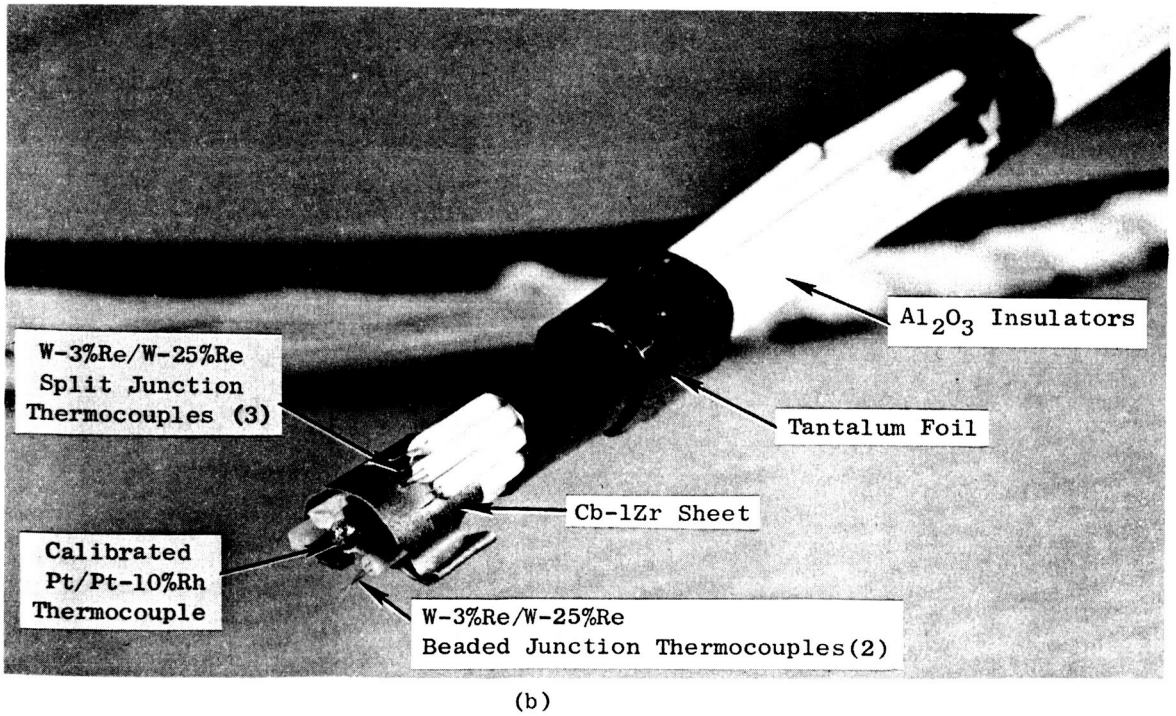
[7] Potassium Corrosion Test Loop Development, Quarterly Progress Report #3, for period ending April 15, 1964, NASA Contract NAS 3-2547, NASA-CR-54091, p. 37.

[8] Potassium Corrosion Test Loop Development, Quarterly Progress Report #4, for period ending July 15, 1964, NASA Contract NAS 3-2547, NASA-CR-54167, p. 63.




(a)




(b)

Figure 12. Components used in the Calibration of the Prototype Loop W-3%Re/W-25%Re Thermocouple Wire. (a) Test Assembly, (b) Enlarged View of Hot Junctions.

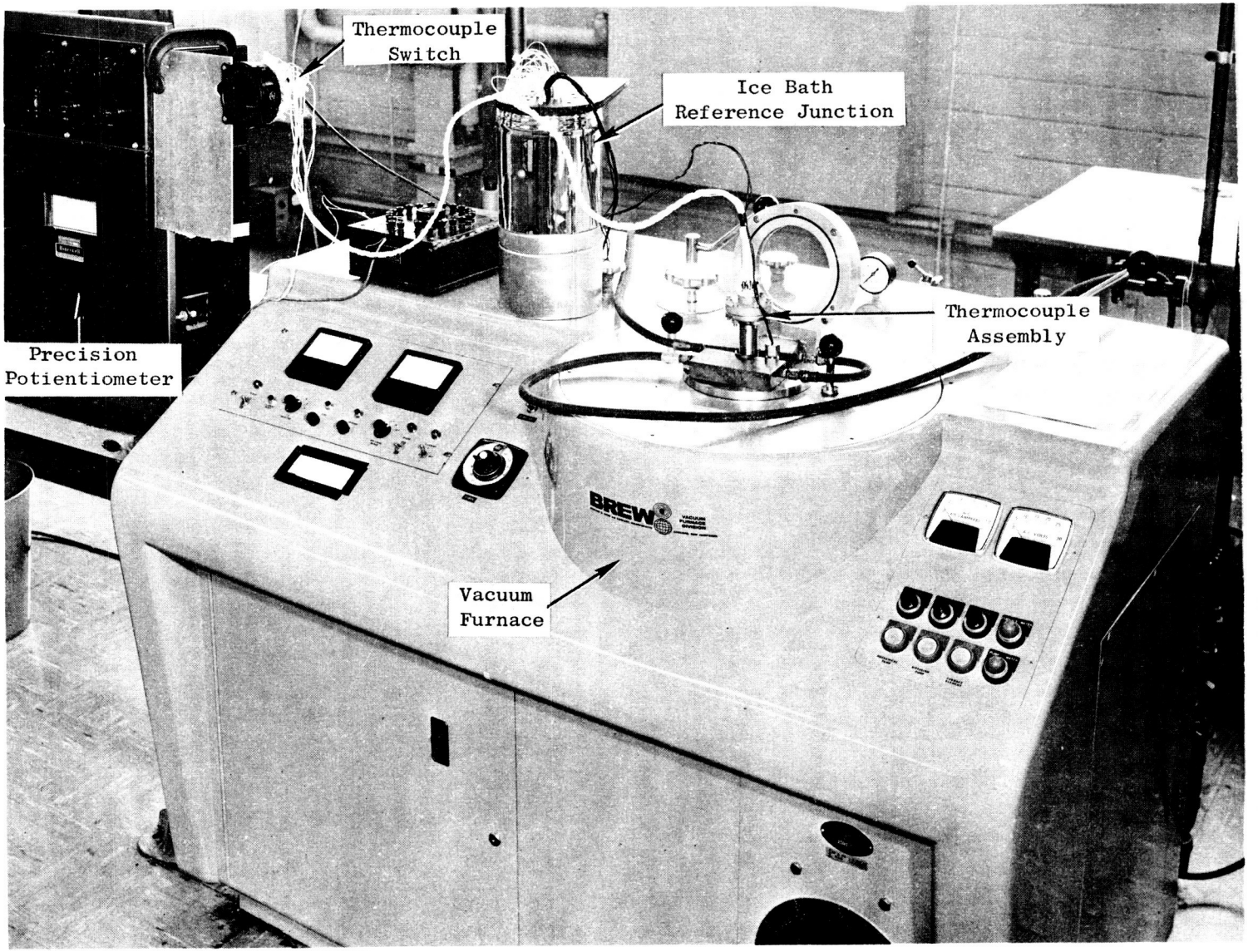



Figure 13. Vacuum Furnace Used in Calibration of W-3%Re/W-25%Re Thermocouple Wire for the Prototype Loop. (C64080609)

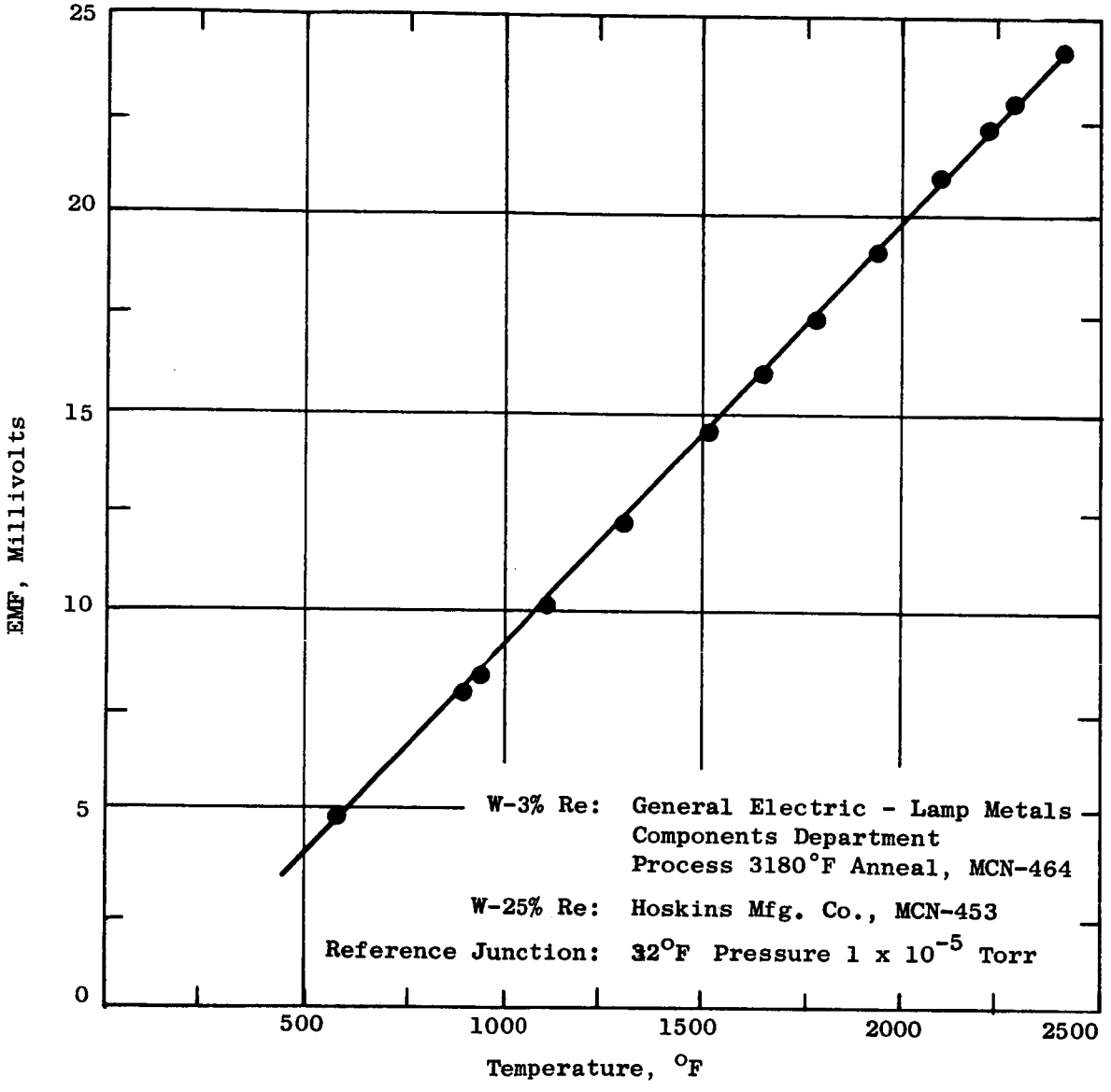



Figure 14. Thermal EMF of W-3% Re/W-25% Re Thermocouple Wire for the Prototype Loop.

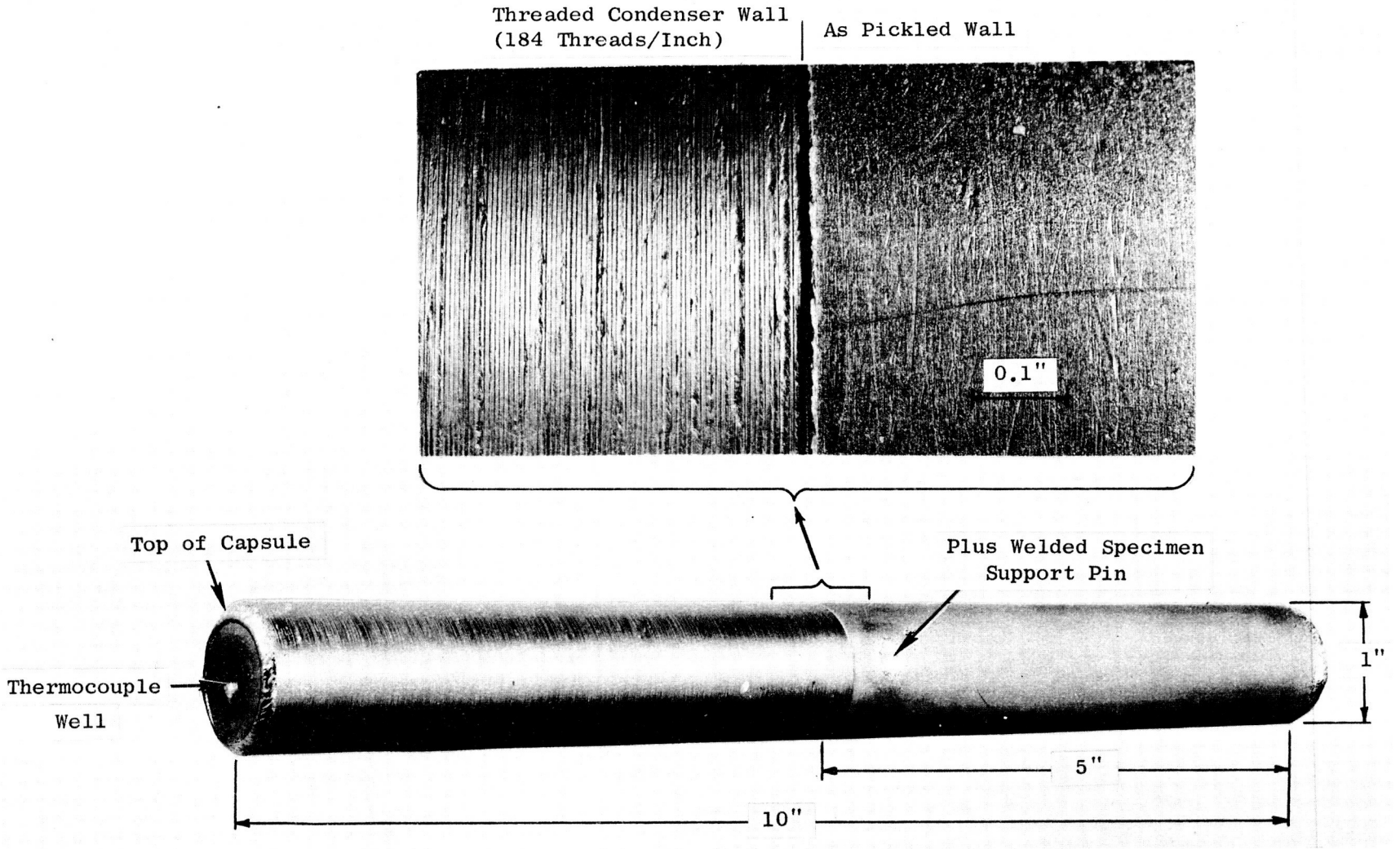



Figure 15. Cb-1Zr Refluxing Potassium Test Capsule Containing Mo-TZM Tubular Inserts in the Condensing Zone. (C6410263-5)

The potassium transfer and capsule sealing was accomplished in the electron beam welding chamber, shown in Figure 16. The unit incorporates facilities for transferring potassium directly from the hot trap to the capsules in a vacuum environment of $5 \times 10^{-5}$ torr. Figure 17 shows an external view of the loading facility with the hot trap in place, and Figure 18 shows the general capsule arrangement inside the chamber. The potassium was transferred to the capsule by pressurizing the hot trap, filling the calibrated stainless steel ladle with the proper amount of potassium and then pouring the potassium through the stainless steel funnel into the capsule using a vacuum rotary feedthrough. After the capsule lid was manually placed on the capsule with the manipulator, the capsule was positioned under the electron beam welding gun at the opposite end end of the chamber by means of the motor driven capsule carriage. The lid was then seal welded to the top of the capsule as the capsule was rotated about its own axis. The second capsule was filled in an identical manner.

The potassium used to fill these capsules was obtained from Mine Safety and Appliance Research Corporation as their high-purity grade. The potassium was vacuum distilled and then hot trapped at 1300°F for 25 hours. An analysis of this potassium at the time of capsule filling is shown in Table VI. A radiographic examination of the capsules showed the welds to be sound and the potassium to be at the proper level.

The refluxing tests will be conducted in the vacuum chamber shown in Figure 19. This system consists of an 18-inch diameter x 30-inch high bakeable chamber connected to a 400 liter/second getter-ion pump. The chamber is capable of reaching the $10^{-10}$ torr range when empty at room temperature. Three cryogenic molecular sieve roughing pumps are used to pump the system down initially. Temperature control will be accomplished by using separately regulated low voltage alternating current power supplies to feed each of the split tantalum strip heaters. Safety circuits have been incorporated into the test facility which will shut off the electric power to the tantalum heaters in the event that the power is lost to the chamber getter-ion pump, the chamber pressure exceeds $5 \times 10^{-5}$ torr, or the temperature of the heat exchanger which surrounds the condenser zone of the test capsule exceeds 300°F.

Figure 20 shows the two test capsule furnaces, heat exchangers and associated equipment installed in the vacuum chamber prior to starting the test. The thermistor readout and control shown is used to monitor the inlet and outlet temperature of the water which extracts heat from the Dowtherm "A"* which fills the heat exchangers. Dowtherm "A" was chosen as the fluid to fill the heat exchanger cavity because of its low vapor pressure in the temperature range of interest, 200° to 300°F. One of the heat exchangers which surrounds the condenser region of the refluxing capsule is shown in Figure 21 prior to installation in the test facility. The internal surface of the heat exchanger was threaded to increase its absorptivity. Following welding of the water lines to the vacuum feedthrough tubes, thermistors were positioned inside the water lines at the inlet and outlet positions of each heat exchanger in order to permit accurate measurement of the temperature rise of the water. A control thermistor located in the well, shown in Figure 21, will shut off electrical power to the capsule furnace if the Dowtherm

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* Dow Chemical Company, Midland, Michigan

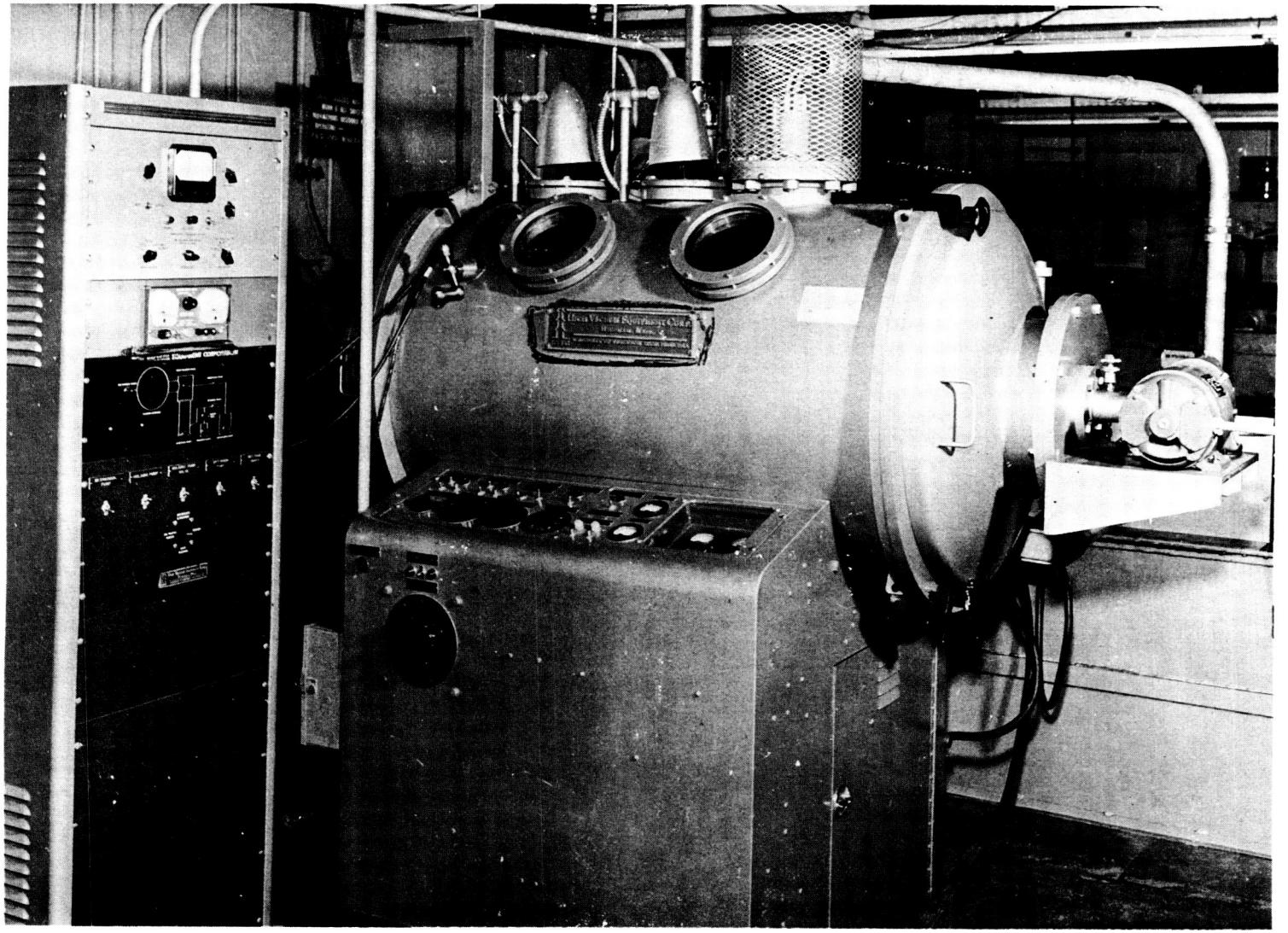

Figure 16. Electron Beam Welding Chamber in Which Refluxing Potassium Capsules were Vacuum Loaded and Sealed by Electron Beam Welding. (C1033002)

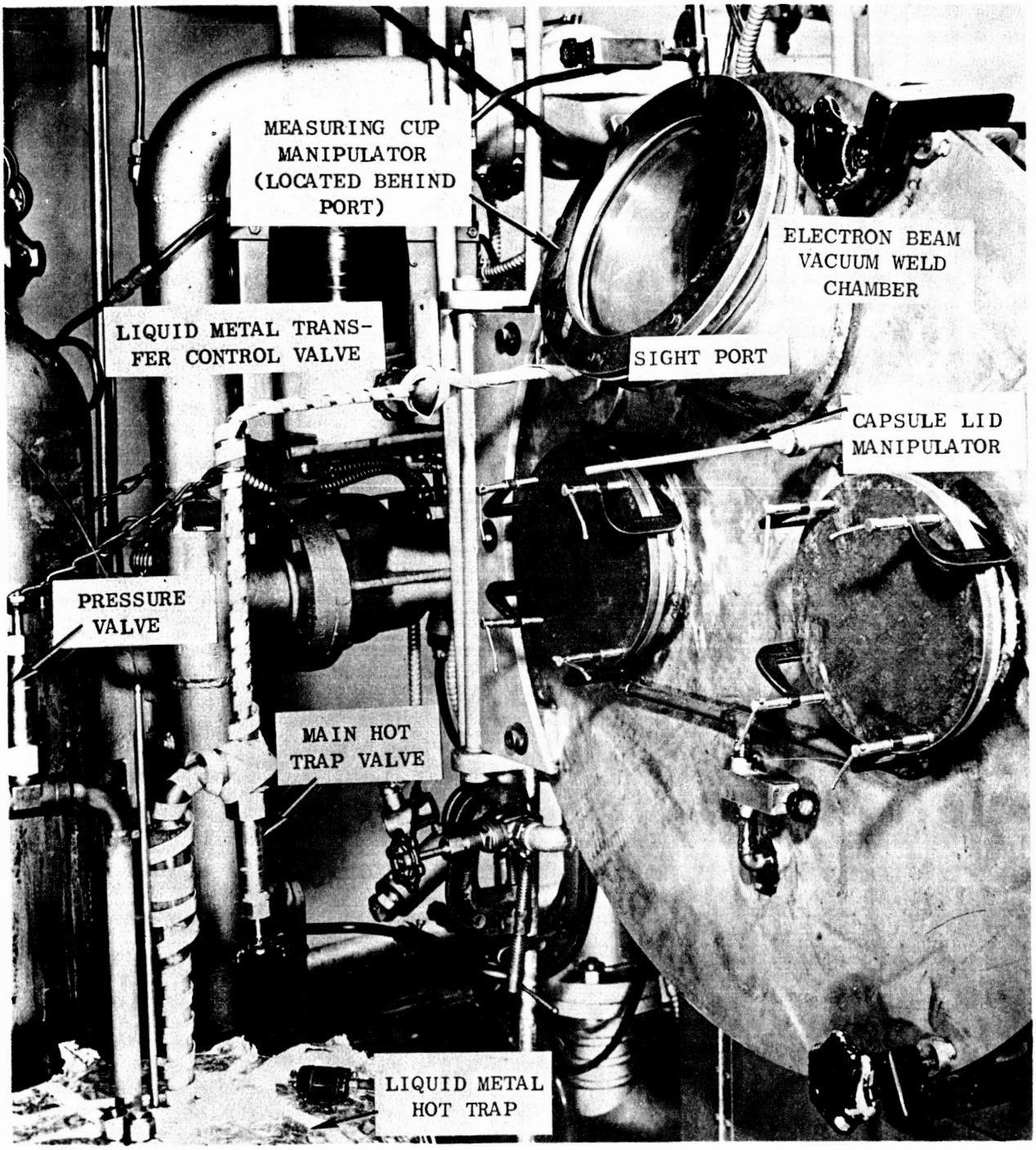


Figure 17. External View of the Apparatus Used in Loading the Refluxing Capsules with Potassium in a Vacuum. Following Loading the Capsules were Sealed by Electron Beam Welding in this Chamber. (C63041623)

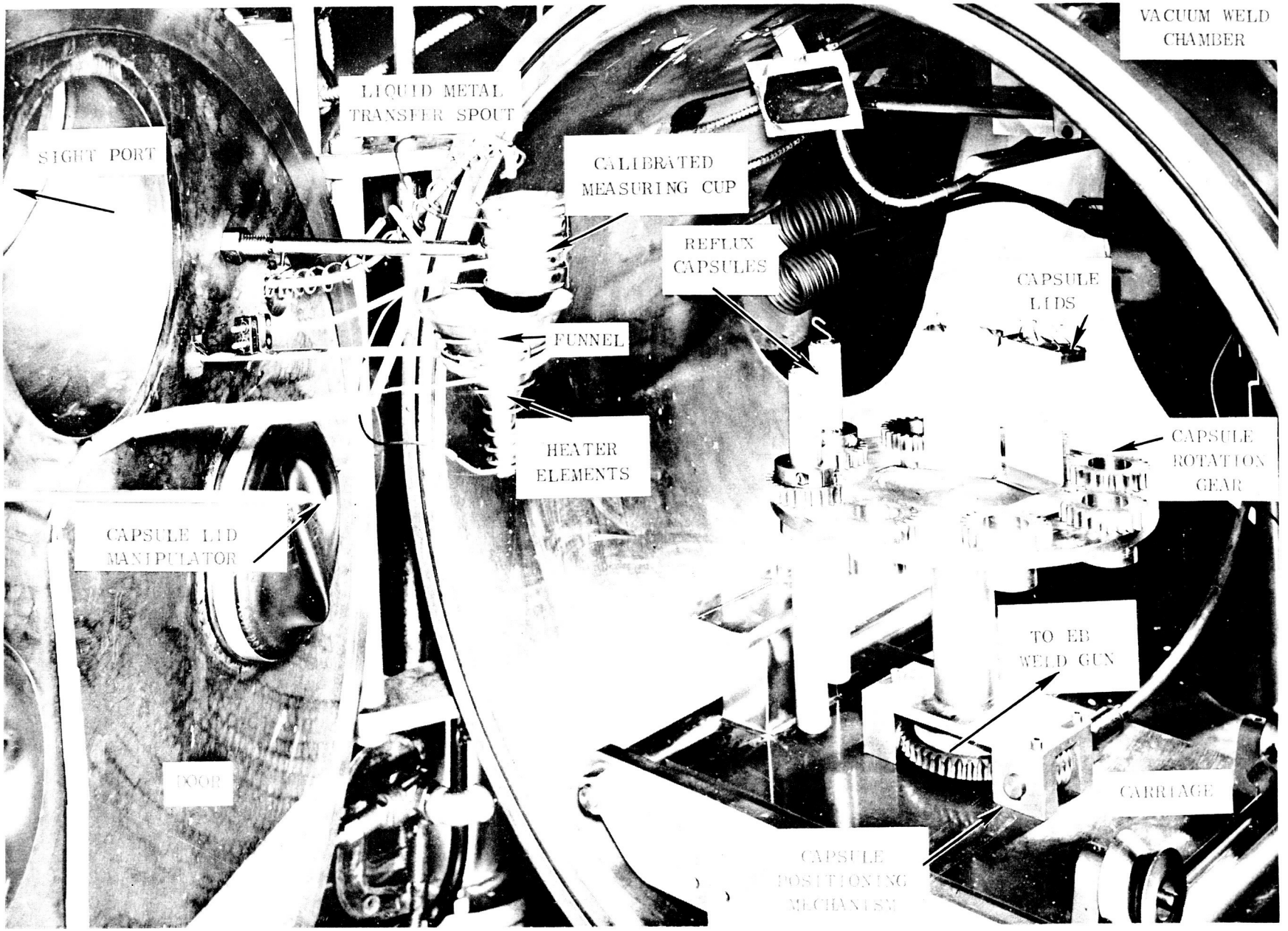


Figure 18. Internal View of the Capsule Loading Facility with the Chamber Door Open. Following Loading of the Capsules with Potassium the Carriage is Moved to the Electron Beam Welding Station at the Other End of the Chamber. (C63041622)

TABLE VI

CHEMICAL ANALYSIS OF POTASSIUM USED TO FILL

Cb-1Zr REFLUXING CAPSULE TESTS

| Sample Identification | Chemical Analysis, ppm(1) | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | O | Fe | Co | Mn | Al | Mg | Sn | Cu | Pb | Cr |
| Capsules I & II | 8.7 | <1 | <1 | <1 | <1 | <1 | <1 | <1 | <1 | <1 |
| | 11.7 | | | | | | | | | |
| Average | 10.2 | | | | | | | | | |

| Sample Identification | Chemical Analysis, ppm(1) | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | Si | Ti | Ni | Mo | Ag | Zr | Ca | Na | Cb |
| Capsules I & II | <5 | <1 | <1 | <1 | <1 | <5 | <1 | <5 | <1 |

(1) Oxygen analysis determined by mercury amalgamation technique as $K_2O$; metallic analyses determined by spectrographic method on KCl.

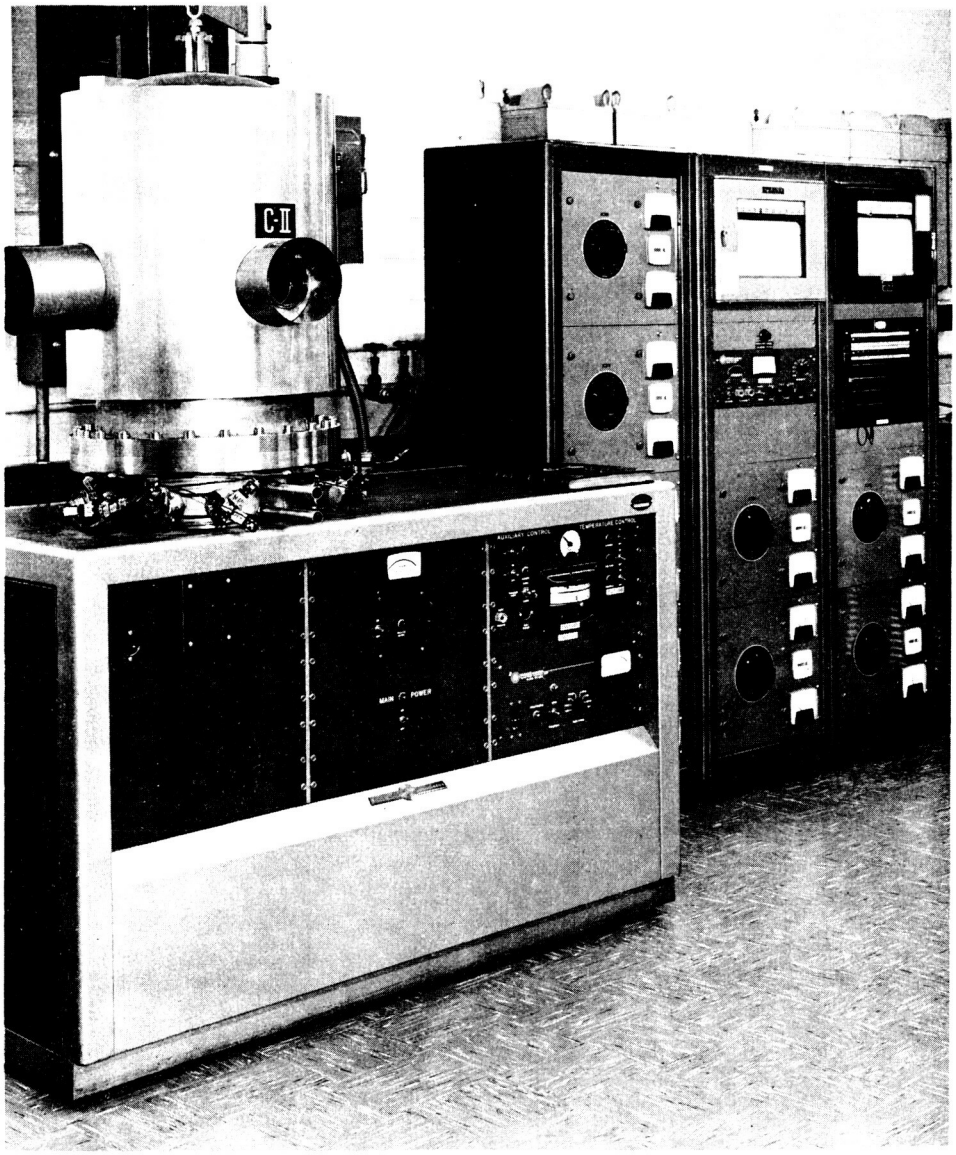


Figure 19. High Vacuum Test Chamber ($10^{-10}$ Torr Range) in Which Cb-1Zr/Mo-TZM Refluxing Potassium Chamber Tests will be Performed. (C64051216)

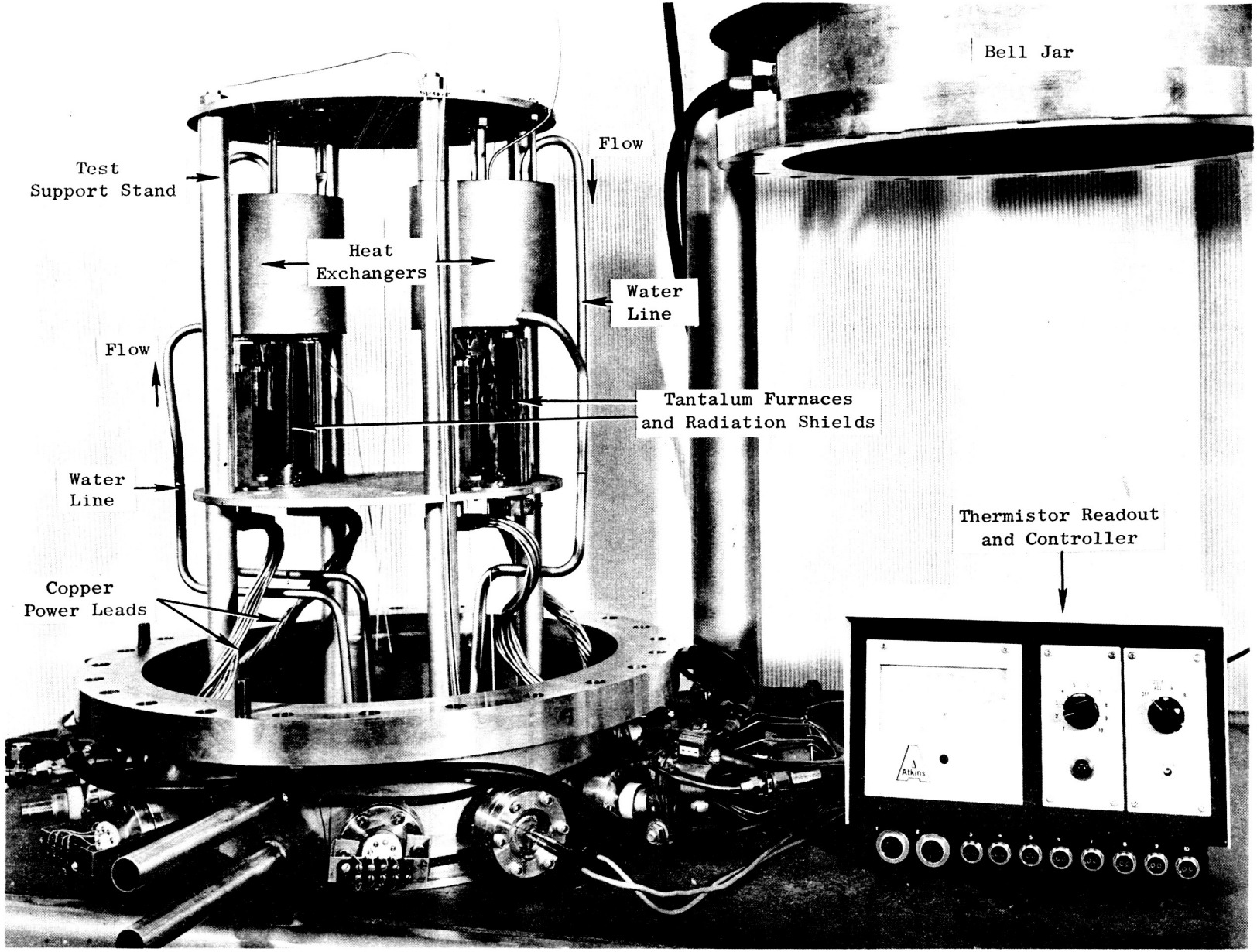


Figure 20. Reflux Corrosion Capsule Test Facility with Bell Jar Removed.
(C64110419)

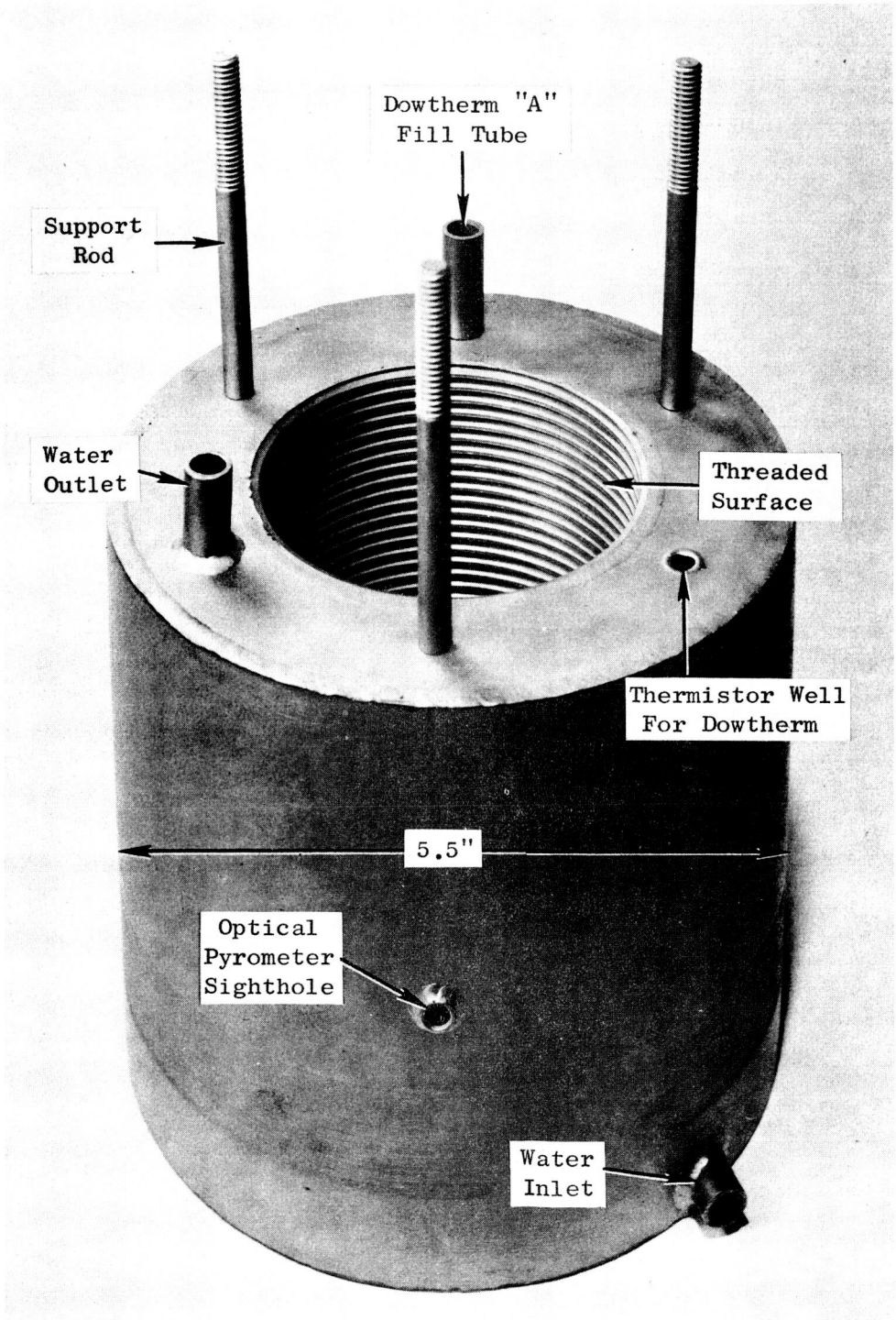


**Figure 21.** Water-Cooled, Dowtherm-Filled Heat Exchanger for the Cb-1Zr/Mo-TZM Refluxing Potassium Test Facility. (C64101924)

temperature exceeds 300°F. Although not anticipated, this could occur if something happened to interrupt the flow of water to the test facility.

The chamber will be baked out and the test started early in November.

In view of the importance of condensing rate in determining the dissolution of refractory alloys on which alkali metal condensation and subsequent washing is occurring, an error analysis has been performed on the methods used to determine the condensing rate in two different types of refluxing potassium compatibility test-systems. The first analysis has been performed on the test method which has been used on a test program[8] which was initiated three years ago at General Electric and the second analysis was performed on the test method described above for evaluating the compatibility of the Mo-TZM alloy. In the previous study, the heat from the condensing zone of four refluxing capsules is rejected to the vacuum chamber walls.

The total heat radiation rate from the emitter to the receiver generally is determined by using the following equation[9]:

$$Q_{12} = A_1 \sigma \frac{1}{\frac{1}{\epsilon_1} \quad \frac{A_1}{A_2} \left( \frac{1}{\epsilon_2} - 1 \right)} \left( T_1^4 - T_2^4 \right) \qquad (1)$$

where:

$\sigma$ = $0.174\ (10^{-8})$ BTU $hr^{-1}$ $ft^{-2}$ $°F^{-4}$

$A_1$ = Area of condensing zone

$A_2$ = Area of chamber receiving radiated heat from capsule

$\epsilon_1$ = Total emittance of capsule

$\epsilon_2$ = Total emittance of vacuum chamber wall

$T_1$ = Absolute temperature of radiating capsule surface, °R

$T_2$ = Absolute temperature of receiver (vacuum chamber surface), °R

The above equation is only valid for concentric spheres or infinite coaxial cylinders. In the earlier tests, four capsules were tested simultaneously in the vacuum chamber and the required condition of coaxial cylinders was not meet. The

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8 Carlson, R. G., et al, Evaluation of a High Strength Columbium Alloy (AS-55) for Alkali Metal Containment, Interim Report Covering the Period November 25, 1961 to July 25, 1962, NASA Contract NAS 3-2140, GE62FPD65, p. 123.

9 Jakob, M. and Hawkins, G. A., Elements of Heat Transfer, John Wiley and Sons, Inc., New York, 1957, p. 230.

error involved by not using a configuration correction factor for non-coaxial cylinders is unknown. Tantalum reflective foil separators used between capsules and re-radiation to the capsules from the separators complicates the analysis. The geometry of the system is too complex for an accurate determination of the errors envolved. The total emittance values may be considered accurate within ± 10% at best, unless elaborate experiments are performed for the determination of the emittance on each alloy, with careful attention to surface condition and environmental history. The accuracy of a brightness pyrometer for determining $T_1$ is limited by the knowledge of the spectral emittance for the alloy and its surface condition. The accuracy of W-3%Re/W-25%Re thermocouples on the surface of the capsule is limited by the calibration of the thermocouples and thermal conduction induced errors at the junction. The accuracy of the temperature measurement, $T_1$, may be considered to be no better than ± 1% at 2000°F.

Using Equation (1) and assuming:

1. Infinite coaxial cylinders and
2. $(T_1^4 - T_2^4) \cong T_1^4$, since

   For case: $T_1 = 2460°R$, $T_2 = 700°R$; $(T_1^4 - T_2^4) = 36.36 \times 10^{12}\ °R^4$

   For case: $T_1 = 2460°R$, $T_2 = 0°R$; $(T_1^4 - T_2^4) = 36.70 \times 10^{12}\ °R^4$

and letting

$A_1 = 0.0327\ ft^2 \pm 2\%$ = Area of 1.5-inch wide radiating band of 1.0-inch OD capsule

$A_2 = 0.589\ ft^2 \pm 2\%$ = Area of 1.5-inch wide receiving band on the 18-inch ID vacuum chamber wall

$\epsilon_1 = 0.5 \pm 10\%$

$\epsilon_2 = 0.3 \pm 10\%$

$T_1 = 2000°F = 2460°R \pm 1\%$

for normal values

$Q_{12} = 978$ BTU/hr

or per unit area of capsule wall

$q_{12} = 29{,}900\ BTU/hr/ft^2$

For reflux rate W (lbs/hr/ft$^2$) of potassium in capsule:

$$W = \frac{q_{12}}{H_v} \qquad (2)$$

where $H_v$ (heat of vaporization) = 674 BTU/lb for K at 2460°R[10]

$$W = 44.4 \text{ lbs/hr/ft}^2$$

Using errors shown and calculating for a maximum $Q_{12}$,

$$Q_{12} = 1{,}150 \text{ BTU/hr}$$

then per unit area of capsule wall

$$q_{12} = 34{,}600 \text{ BTU/hr/ft}^2$$

and the

$$\text{Error (max)} = \frac{(q_{12})_{\text{max}} - (q_{12})_{\text{normal}}}{(q_{12})_{\text{normal}}} \times 100\% \qquad (3)$$

$$\text{Error (max)} = 15.7\%$$

This calculated error value is considered to be an optimistic minimum in view of the uncertainty concerning the emittance and the failure to rigorously satisfy geometry requirements in practical test systems.

For the water cooled heat exchanger facility which will be used in the current study, the total heat radiated is determined by measuring only the flow rate of the cooling water and the inlet and outlet water temperatures at the heat exchanger. Water flow-measurements are determined by weighing volumes collected in a ten-minute period. Because of the relatively low temperatures of the water, its temperature rise may be accurately measured with the thermistors positioned at the inlet and outlet locations in the water lines of the heat exchanger. It is assumed that all the heat radiated from the capsule is transferred to the cooling water. The facility has been designed so as to reduce heat losses to other regions to an insignificant level.

The total heat radiation rate from the capsule to the heat exchanger is then calculated from the equation:

$$Q_{12} = C_p \, m \, (\Delta T) \qquad (4)$$

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10 Lemon, A. W., Jr., et al, Engineering Properties of Potassium, Final Report Battelle, NASA CR-54017.

where

$C_p$ = heat capacity of cooling water, average of values for inlet and outlet temperature, BTU/lb/°F,

m = weight of cooling water per unit time,

$\triangle T$ = temperature difference of inlet and outlet cooling water, °F.

let

$C_p$ = 1 (accurate values for $C_p$ as a function of temperature can be found in Tables.)

m = 29.3 lbs/hr ± 1%

$\triangle T$ = 100°F ± 2%

and for normal values using equation (4)

$Q_{12}$ = 2,930 BTU/hr

or per unit area of capsule wall

$q_{12}$ = 29,900 BTU/hr/ft$^2$ (total radiating area = 0.098 ft$^2$)

Using equation (2)

W = 44.4 lbs/hr/ft$^2$

Using errors shown and calculating for a maximum $Q_{12}$

$Q_{12}$ = 3,008 BTU/hr

and per unit area of capsule wall

$q_{12}$ = 31,000 BTU/hr/ft$^2$

from equation (3)

Error (max) = 3.7%

The increased accuracy and simplicity of the reflux rate determinations by using the water cooled heat exchanger is easily seen from these calculations and their assumptions. A similar method of determining the reflux rate in alkali metal compatibility tests is used in studies currently in progress at the Oak Ridge National Laboratory[11].

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[11] Personal Communication from W. C. Thurber, Oak Ridge National Laboratory.

## 8. Grain Growth Studies on Cb-1Zr Alloy

A limited investigation to determine the susceptibility of Cb-1Zr alloy to abnormal grain growth following cold working and annealing has been completed. The results of earlier forming and annealing studies on Cb-1Zr alloy tubing used in the fabrication of Loops I and II of this program have been reported[12]. A marked tendency to undergo grain coarsening or abnormal grain growth was observed in the lot of tubing used in the construction of Loop I; the lot of tubing for Loop II and the Prototype Loop exhibited little or no grain coarsening. It was previously suggested that the difference in behavior may have been due to the lower interstitial element concentration in the Loop I tubing (total of 169 ppm vs 262 ppm). An additional series of grain growth experiments have been conducted on cold worked bars of Cb-1Zr alloy and the results of this work are reported below.

Samples of Cb-1Zr alloy bar*, 1-inch thick x 0.5-inch wide x 4-inch long, were machined into wedge-shaped specimens tapering from 0.750-inch to 0.062-inch thick. These wedges were press forged at room temperature to approximately 0.180-inch thick resulting in a deformation gradient in the material of 0 to 76%. The sketch of one of the wedge specimens which illustrates the method of specimen preparation is given in Figure 22. Each forging was sectioned along its longitudinal axis, pickled in a solution of 20%HF-20%$HNO_3$-60%$H_2O$, and sequentially rinsed in water and ethyl alcohol. Then, sectioned lengths were annealed in vacuum (1 x $10^{-5}$ torr) for one hour and 100 hours at each of the following temperatures and examined metallographically: 2000°, 2200°, and 2400°F, Prior to preparation of the cut surface for metallographic examination, approximately 0.062 inch was removed from the surface to eliminate any structural changes resulting from the sectioning of the forging before the annealing cycle. Micrographs representing the cold work gradient in the as-forged specimens and the effects of annealing are depicted in Figure 22. The micrographs are representative of the appearances of areas in each specimen having approximately the amount of cold work indicated by the arrows. It should be noted that no cold work is evident in the first two micrographs, A-1 and A-2. Although no reduction in thickness was achieved here as a result of the initial geometry of the specimens, some induced strain from lateral flow is possible. Examination of Series B, samples forged and annealed for one hour at 2200°F, indicates that recrystallization occurred in Specimens B-3 through B-8 to produce a fairly uniform grain size. This is also the case for specimens C-3 through C-8 which represent material forged and annealed for one hour at 2400°F. The larger grain size depicted in Specimen C-2 is not thought to be a result of recrystallization and subsequent grain growth but, rather, a reduction in grain boundary area of the primary recrystallized grains, influenced by small amounts of induced strain from forging.

While this experiment did not reveal a level of critical strain which would correspond directly to the abnormal grain growth results obtained on bending and annealing Cb-1Zr alloy tubes, it did indicate that grain coarsening or possibly

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12 Potassium Corrosion Test Loop Development, Quarterly Progress Report #3, Covering the period January 15, 1964 to April 15, 1964, NASA Contract NAS 3-2547, NASA-CR-54081, p. 40.

* Stellite Division of UCC, Heat 5155; Chemical Analysis (5): 1.09 Zr, 0.0018 O, 0.0013 N, 0.0020 C, 0.0001 H.

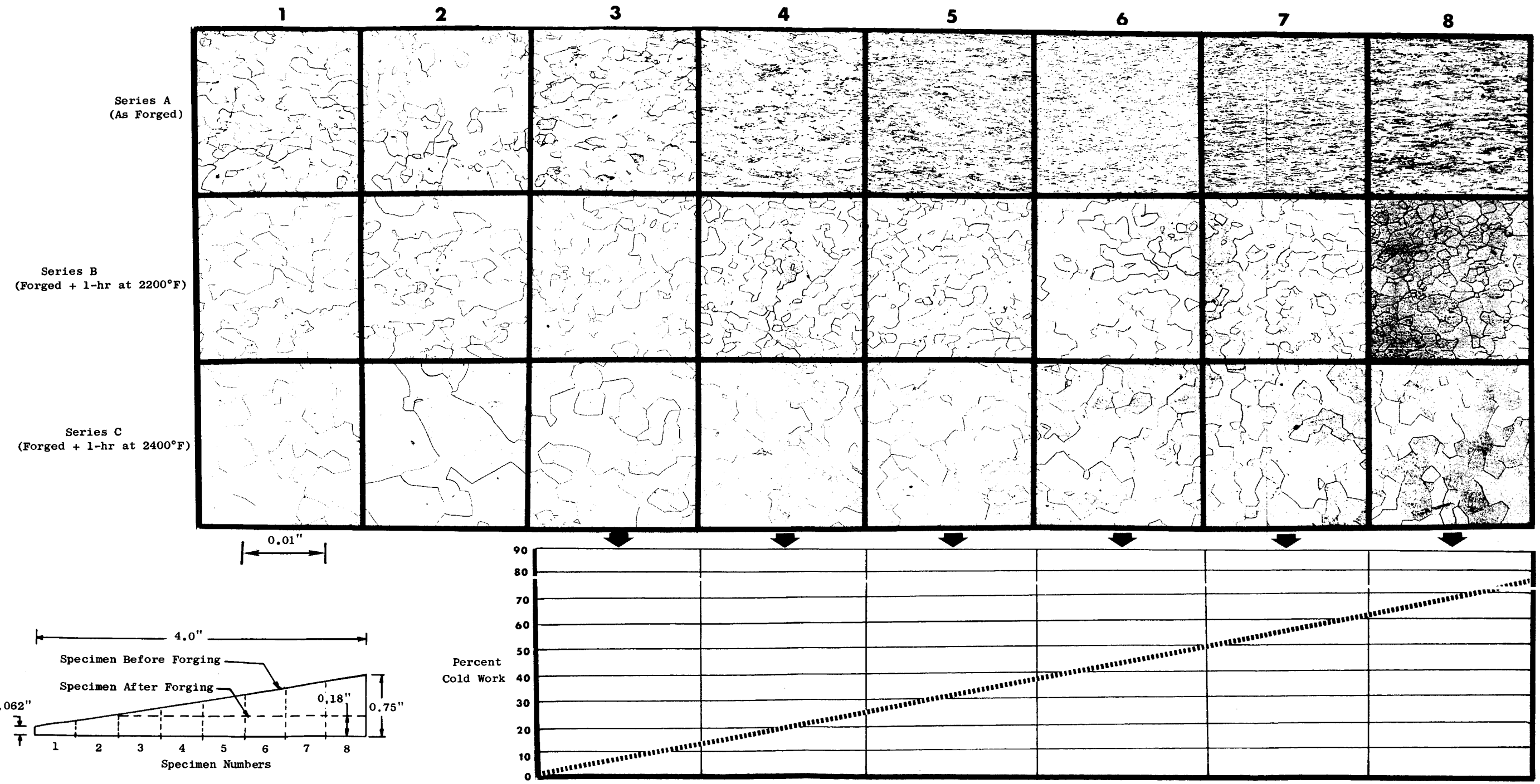


Figure 22. Isochronal Heat Treatment Survey Showing the Grain Size of Cb-1Zr Specimens as a Function of Percent Cold Work and Annealing Temperature.

abnormal grain growth can occur in the Cb-1Zr alloy under specific conditions without primary recrystallization. The fact that a critical strain level was not indicated in this experiment is somewhat disconcerting. This is particularly true since the total interstitial content of the Cb-1Zr alloy used in this experiment is lower (52 ppm) than the total interstitial content of the Cb-1Zr alloy used in the previous work (169 ppm) in which pronounced grain coarsening was observed. The primary difference between the two lots of material is the method used in inducing strain (forging vs bending). It is evident that additional experimentation is required in order to fully understand the interrelated effects of the amount of strain, strain gradient, method of deformation, and chemistry on the recrystallization and grain growth behavior of Cb-1Zr alloy.

9. Helium Analysis System

Cylinder helium is now on hand having an analyzed impurity content not exceeding 2 ppm $O_2$ and 2 ppm $H_2O$. As of July 27, 1964, this helium has been used for all welding on the corrosion loop program. The purification train continues to be used to purify the gas further before admission to the welding chamber.

Reliable analyses of helium at impurity levels of a few ppm are now being obtained by calibrating the partial pressure analyzer against helium of known impurity content prior to analysis of the unknown sample*. The procedure used is to first obtain a "background" spectrum from ultra-high purity helium. A spectrum of the calibrating gas is then obtained. The sensitivity factor for each impurity is then obtained from the increase in ion current at the appropriate mass to charge ratio of the calibrating gas over the "background" gas. These sensitivity factors are then used to calculate the impurity content from the spectrum of the unknown sample gas.

Analyses of both the calibrating gas and the ultra-pure helium have been obtained from the vendor. In addition, oxygen analysis of the calibrating mixture has been obtained here using a sensitive Brady apparatus. The results of these analyses are given in Table VII. The Brady analysis is in good agreement with the vendor analysis.

In order to conveniently introduce the various gases into the analysis system, the sample lines have been modified and a trapped mechanical pump has been installed so that the sample line may be evacuated to about 1 micron.

A photograph of the present system is shown in Figure 23. The gas cylinder rack in the right foreground holds cylinders containing the ultra-pure helium and the calibrating mixture. These gases are introduced into the sample line through the valves mounted on top of the rack. The partial pressure analyzer and vacuum system are shown in the center background, and the associated electronic equipment is on the cart in the left foreground.

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* Analysis of the water vapor cannot be obtained in this way, and it is currently being determined with a dew point cup. In the future, an electrolytic hygrometer will also be used for water vapor determinations.

TABLE VII

ANALYSES OF ULTRA PURE HELIUM AND CALIBRATING MIXTURE

| | Ultra-Pure Helium Vendor Analysis, ppm | Calibrating Mixture Vendor Analysis, ppm | Calibrating Mixture Brady Analysis, ppm |
|---|---|---|---|
| $O_2$ | 0.1 | 17 | 18.3 |
| $N_2$ | 0.3 | 12 | |
| $H_2$ | 0.2 | 20 | |
| CO | 0.0 | 15 | |
| $CO_2$ | 0.1 | | |
| Ne | 10.9 | | |
| A | 0.0 | | |
| $CH_4$ | 0.0 | | |
| $H_2O$ | 2.5 | | |
| He | Bal | Bal | |

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| $N_2$ | 0.3 | 12 | |
| $H_2$ | 0.2 | 20 | |
| CO | 0.0 | 15 | |
| $CO_2$ | 0.1 | | |
| Ne | 10.9 | | |
| A | 0.0 | | |
| $CH_4$ | 0.0 | | |
| $H_2O$ | 2.5 | | |
| He | Bal | Bal | |

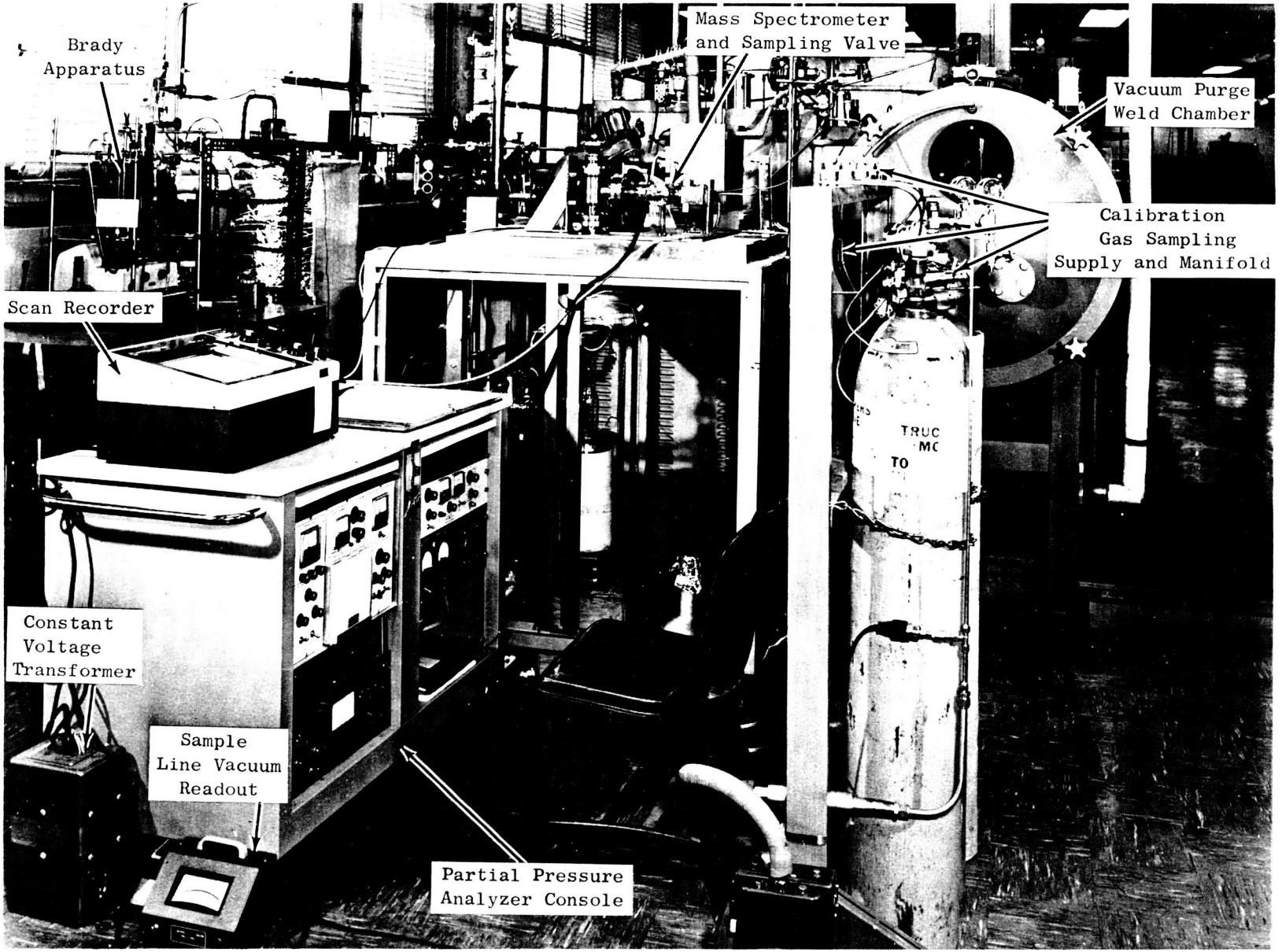


Figure 23. Partial Pressure Gas Analyzer and Sampling System Used to Monitor Purity of Helium Gas in Vacuum Purge Welding Chamber. (C64102071)

## III FUTURE WORK

A. The Loop II test will be terminated on November 6 after completion of the 2,500-hour test. An additional 100-hour test will be conducted on this system following repair of thermocouples, pressure transducer leads and the metering valve actuation systems.

B. The sodium will be discharged from Loop II and the residual sodium will be vacuum distilled from the loop.

C. Fabrication of Prototype Loop components and the associated alkali metal purification and handling system will continue.

D. Calibration and checkout tests on available Prototype Loop components will be performed.

E. The refluxing potassium compatibility tests (Mo-TZM inserts in Cb-1Zr capsules) will be started early in the next quarter.

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